(2)

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

WASHINGTON, D.C. 20460

OPP OFFICIAL RECORD
HEALTH EFFECTS DIVISION
SCIENTIFIC DATA REVIEWS
EPA SERIES 361

OFFICE OF
PREVENTION, PESTICIDES
AND TOXIC SUBSTANCES

MEMORANDUM

Date: 1/12/05

Subject: Mesotrione. Summary of Analytical Chemistry and Residue Data for Sweet Corn, PP#2F06443, and Response to Data Deficiencies of a Previous HED Review (PP#8F04954, DP Barcodes: D245477 and D260267, 6/6/01, S. Levy).

DP Number:	D283827	Decision Number:	208903
PC Code:	122990	MRID Numbers:	45651801-45651803, 45651813, 45651814, 45651816, 45651817, 45665901
40 CFR 180.	571	Chemical Class:	triketone herbicide

From: William Cutchin, Chemist
Technical Review Branch
Registration Division (7505C)

Through: George Kramer, Chemist
Registration Action Branch 1
Health Effects Division (7509C)

To: J. Stone/ J. Miller PM 23
Herbicide Branch
Registration Division (7505C)

This document was originally prepared under contract by Dynamac Corporation and has been reviewed by TRB and revised to reflect current OPP policies.

Executive Summary

Syngenta Crop Protection has submitted a petition for the establishment of permanent tolerances for residues of the herbicide mesotrione (2-[4-(methylsulfonyl)-2-nitrobenzoyl]-1,3-cyclohexanedione; designated by the company code ZA1296) on sweet corn. The petitioner is proposing the establishment of permanent tolerances for residues of mesotrione in/on the following commodities:

Sweet corn grain (K+CWHR)	0.01 ppm
Sweet corn forage	0.50 ppm
Sweet corn stover	2.0 ppm

MAR - 1 2005 +1.5

Syngenta has submitted crop field trial data (45651801) and a summary document for the calculation of livestock dietary burdens as a result of the new use (45651817) to support the proposed tolerances.

Mesotrione was the subject of a previous petition (PP#8F04954, DP Barcodes: D245477 and D260267, 6/6/01, S. Levy) for field corn uses in the U.S. The current petition amends the 4 lb/gal flowable concentrate (FlC) formulation to include uses on sweet corn. In the previous petition, HED recommended for the proposed field corn uses and tolerances, with a conditional registration until submission of the following additional data:

- storage stability data for the [cyclohexane-^{14}C]mesotrione corn and goat metabolism studies;
- a revised interference study;
- a successful petition method validation (PMV) of the analytical method;
- revised Sections B and F.

Syngenta has also submitted data to address the requirements of the previous petition (MRIDs 45651802, 45651803, 45651813, 45651814, 45651816, and 45665901).

Currently, there are four end-use products containing mesotrione registered for food/feed use: a 4 lb/gal FlC formulation (EPA Reg. No. 100-1131) and three multiple active ingredient formulations, a 0.29 lb/gal emulsifiable concentrate (EC) formulation (with acetochlor; EPA Reg. No. 100-1142), a 0.33 lb/gal EC formulation (with S-metolachlor; EPA Reg. No. 100-1148), and a 0.268 lb/gal EC formulation (with atrazine and S-metolachlor; EPA Reg. No. 100-1152). These products are registered for use on field corn and yellow popcorn. The petitioner is proposing to add sweet corn uses to the labels for the 4 lb/gal FlC, 0.33 lb/gal EC, and 0.268 lb/gal EC formulations. The 4 lb/gal FlC formulation is to be applied to sweet corn as a maximum of two broadcast preemergence or postemergence applications at a total application rate of 0.34 lb ai/A with a 45-day PHI. The EC formulations are to be applied preplanting at up to 0.20 lb ai/A.

Tolerances for residues of mesotrione are currently established for field corn forage, grain, and stover and popcorn grain and stover, each at 0.01 ppm [§180.571(a)]. Section 18 emergency exemption time-limited tolerances, which expire 06/30/04, are established for residues of mesotrione in/on sweet corn kernel plus cob with husks removed (0.01 ppm), forage (0.50 ppm), and stover (2.0 ppm) [§180.571(b)]. The HED Metabolism Assessment Review Committee (MARC) has concluded (PP#8F04954, DP Barcode: D274111, 4/26/01, S. Levy) that for tolerance expression and risk assessment purposes, the residue of concern in/on field corn, livestock, and rotational crop commodities is mesotrione *per se*.

Crop field trial data have been submitted for sweet corn reflecting the proposed use pattern for the 4 lb/gal FlC formulation; however, additional information regarding adjuvants used in the field trials is required. No feeding study data have been submitted; it has been determined that feeding study data are not required to support this petition. The existing enforcement method for field corn commodities may be used for sweet corn commodities. The available rotational crop

data indicate that the proposed rotational crop restrictions are appropriate. There are currently no international Codex tolerances established for mesotrione.

Regulatory Recommendations and Residue Chemistry Deficiencies

HED has examined the residue chemistry database for mesotrione. The petitioner has adequately responded to the outstanding data requirements specified in the review of the field corn petition (PP#8F04954, DP Barcodes: D245477 and D260267, 6/6/01, S. Levy). There are now no residue chemistry issues that would preclude unconditional registration of mesotrione on field corn.

Pending resolution of the deficiencies noted below, there are no residue chemistry issues that would preclude granting a conditional registration for mesotrione on sweet corn or establishment of permanent tolerances for mesotrione as follows:

corn, sweet, kernel plus cob with husks removed	0.01 ppm
corn, sweet, forage	0.50 ppm
corn, sweet, stover	1.5 ppm

860.1200 Directions for Use

The petitioner must clarify the rotational crop restriction to define which types of corn (field corn, popcorn, and/or sweet corn) have a 0-day plantback interval; if the petitioner intends to allow a 0-day plantback interval for sweet corn, it should be removed from the list of crops which may be rotated the following season.

Note to PM: The label for the 4 lb/gal FlC formulation indicates tank mix uses on sweet corn with the following products which do not currently have registered uses on sweet corn: Axiom®, Bicep®, Bicep Lite II®, Degree™, Degree Xtra™, Doubleplay®, Dual II®, Fultime™, Harness®, Harness Xtra®, Liberty®, Liberty ATZ, Surpass® EC, and Topnotch®. These tank mixes should be removed.

860.1550 Proposed Tolerances

The proposed tolerance of 2.0 ppm on sweet corn stover is too high; a tolerance of 1.5 ppm is more appropriate. A revised Section F is required.

Background

Mesotrione [2-[4-(methylsulfonyl)-2-nitrobenzoyl]-1,3-cyclohexanedione] is a triketone herbicide which inhibits the enzyme p-hydroxyphenylpyruvate dioxygenase (HPPD), disrupting the pigment biosynthesis in susceptible plants. Mesotrione is intended for preemergence and postemergence use for selective control of annual broadleaf weeds in corn.

Mesotrione was the subject of a previous petition (PP#8F04954, DP Barcodes: D245477 and D260267, 6/6/01, S. Levy) for field corn uses in the U.S. The current petition, amends the 4

lb/gal FlC formulation to include uses on sweet corn. The chemical name and structure for mesotrione is presented below.

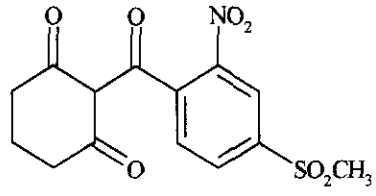
Table 1. Nomenclature of Test Compound	
Chemical structure	
Common name	Mesotrione
IUPAC name	2-(4-mesyl-2-nitrobenzoyl)cyclohexane-1,3-dione
CAS name	2-[4-(methylsulfonyl)-2-nitrobenzoyl]-1,3-cyclohexanedione
CAS registry number	104206-82-8
Company code	ZA1296
Other synonyms	Callisto™

Table 2. Physicochemical Properties of the Technical Grade Test Compound.		
Parameter	Value	Reference
Melting point/range	148.7-152.5 °C	RD Memo, D263245, 2/24/00, H. Podall
pH	3.4 (1% dispersion in water; 25 °C)	RD Memo, D263245, 2/24/00, H. Podall
Density	1.46 g/mL, 20 °C	RD Memo, D263245, 2/24/00, H. Podall
Water solubility	20 ° C 160 ppm, unbuffered water 0.22 g/100 mL, pH 4.8 1.5 g/100mL, pH 6.9 2.2 g/100 mL, pH 9	RD Memo, D263245, 2/24/00, H. Podall
Solvent solubility	20 ° C 0.37 g/100 mL, methanol 1.7 g/100 mL, ethyl acetate 0.27 g/100 mL, toluene 10.4 g/100 mL, acetonitrile <0.03 g/100 mL, heptane 8.1 g/100 mL, acetone	RD Memo, D263245, 2/24/00, H. Podall
Vapor pressure	4.3 x 10^{-8} torr, 20 °C	RD Memo, D263245, 2/24/00, H. Podall
Dissociation constant, pK_a	3.12, 20 °C	RD Memo, D263245, 2/24/00, H. Podall
Octanol/water partition coefficient, Log(K_{OW})	20 ° C log P_{OW} = 0.11 in unbuffered water log P = 0.90 in pH 5 buffer log P <-1 at pH 7 and 9 buffered water	RD Memo, D263245, 2/24/00, H. Podall
UV/visible absorption spectrum	*Absorption maximum in methanol at 256* mu, with a molar extinction coefficient of 2.24 x 10^4 M cm.	*RD Memo, D263245, 2/24/00, H. Podall*

860.1200 Directions for Use

Table 3. Summary of End-Use Products Proposed for Use on Sweet Corn.

Trade Name	Reg. No.	ai (% of formulation)	Formulation Type	Target Crops	Target Pests	Label Date
Callisto™ Herbicide	100-1131	40.0 [4 lb/gal]	Suspension concentrate (FlC)	Field corn, production seed corn, sweet corn and corn grown for silage	Annual broadleaf weeds	N/A
Mesotrione/S-Metolachlor Herbicide [Camix Selective Herbicide]	100-1148	3.68 [0.33 lb/gal]	Emulsifiable concentrate (EC)	Sweet corn	Annual grasses and broadleaf weeds	N/A
Mesotrione/S-Metolachlor/ Atrazine Herbicide [Lumax Selective Herbicide]	100-1152	2.94 [0.268 lb/gal]	EC	Sweet corn	Annual grasses and broadleaf weeds	N/A

Table 4. Summary of Directions for Use of Mesotrione on Corn.

Formulation [EPA Reg. No.]	Applic. Timing, Type, and Equip.	Applic. Rate (lb ai/A)	Max. No. Applic. per Season	Max. Seasonal Applic. Rate (lb ai/A)	PHI (days)	Use Directions and Limitations
Corn (including field, sweet, production seed corn, and corn grown for silage) [1]						
4 lb/gal FlC [100-1131]	Preemergence Soil Broadcast Ground	0.188-0.24	2 [2]	0.34 [3]	45 (forage, grain, stover)	Postemergence applications may be made to corn up to 30 inches tall or up to the 8-leaf growth stage. A minimum retreatment interval of 14 days is proposed.
	Postemergence Foliar Broadcast Ground/aerial	0.094				
Sweet corn						
0.33 lb/gal EC [100-1148]	Preplant [type and equipment not specified]	0.13-0.20	Not specified	Not specified	Not specified	Application rates are dependent on soil texture and organic matter content, with highest rate to be applied to fine soils with ≥3% organic matter. Application is to be made within 10 days prior to planting.

Table 4. Summary of Directions for Use of Mesotrione on Corn.

Formulation [EPA Reg. No.]	Applic. Timing, Type, and Equip.	Applic. Rate (lb ai/A)	Max. No. Applic. per Season	Max. Seasonal Applic. Rate (lb ai/A)	PHI (days)	Use Directions and Limitations
0.268 lb/gal EC [100-1152]	Preplant [type and equipment not specified]	0.10-0.20	Not specified	Not specified	Not specified	Application rates are dependent on soil texture and organic matter content, with highest rate to be applied to fine soils with ≥3% organic matter. Application is to be made within 10 days prior to planting.

[1] Use on popcorn or ornamental (Indian) corn is prohibited.
[2] A single preemergence and single postemergence application or two postemergence applications may be made.
[3] Maximum seasonal rate for preemergence plus postemergence applications. A maximum seasonal rate of 0.24 lb ai/A is proposed for preemergence applications alone and a maximum seasonal rate of 0.188 lb ai/A is proposed for postemergence applications alone.

The tank mix recommendations for the specimen label for the 4 lb/gal FlC formulation are detailed in the table below.

Table 5. Recommended Tank Mixtures

Product	Active ingredient	Established Use on Sweet Corn
Preemergence applications		
2,4-D	2,4-D	Yes
AAtrex®	atrazine	Yes
Axiom®	metribuzin + flufenacet	No; flufenacet may currently only be used on field corn
Bicep®	atrazine + metolachlor	No; product cancelled
Bicep Lite II®	atrazine + metolachlor	No; product cancelled
Bicep II Magnum®	atrazine + S-metolachlor	Yes
Bicep Lite II Magnum®	atrazine + S-metolachlor	Yes
Degree™	acetochlor	No; acetochlor may currently only be used on field corn
Degree Xtra™	acetochlor + atrazine	No; acetochlor may currently only be used on field corn
Doubleplay®	acetochlor + EPTC	No; acetochlor may currently only be used on field corn
Dual II®	metolachlor	No; product cancelled
Dual II Magnum®	S-metolachlor	Yes
Dual Magnum®	S-metolachlor	Yes
Frontier®	dimethenamid	Yes
Fultime™	acetochlor + atrazine	No; acetochlor may currently only be used on field corn

Table 5. Recommended Tank Mixtures		
Product	**Active ingredient**	**Established Use on Sweet Corn**
Gramoxone® Extra	paraquat dichloride	Yes
Gramoxone Max	paraquat dichloride	Yes
Guardsman®	atrazine + dimethenamid	Yes
Guardsman Max®	atrazine + dimethenamid-P	Yes
Harness®	acetochlor	No; acetochlor may currently only be used on field corn
Harness Xtra®	acetochlor + atrazine	No; acetochlor may currently only be used on field corn
LeadOff™	atrazine + dimethenamid	Yes
Outlook™	dimethenamid-P	Yes
Prowl®	pendimethalin	Yes
Roundup Ultra™	glyphosate	Yes
Roundup UltraMax™	glyphosate	Yes
Surpass® EC	acetochlor	No; acetochlor may currently only be used on field corn
Topnotch®	acetochlor	No; acetochlor may currently only be used on field corn
Touchdown®	glyphosate-trimesium (sulfosate)	Yes
Postemergence applications		
AAtrex 4L	atrazine	Yes
AAtrex Nine-O	atrazine	Yes
Basagran	bentazon	Yes
Liberty®	glufosinate-ammonium	No; glufosinate-ammonium may currently only be used on field corn
Liberty ATZ	glufosinate-ammonium + atrazine	No; glufosinate-ammonium may currently only be used on field corn

The proposed label for the 4 lb/gal FlC formulation states that crop yield loss may occur: (i) if postemergence application of mesotrione is made to corn treated with Counter (terbufos) or Lorsban (chlorpyrifos); (ii) if mesotrione is tank-mixed with any organophosphate or carbamate insecticide as a postemergence application; or (iii) if any organophosphate or carbamate insecticide is applied within 7 days before or after application of mesotrione to corn.

For the 4 lb/gal FlC formulation, application through any type of irrigation system is prohibited, and applications are not to be made with suspension fertilizers or emulsifiable concentrate grass herbicides (postemergence). Applications are to be made in a minimum spray volume of 10 gal/A using ground equipment or 3 gal/A for aerial equipment. Preemergence applications are to be made with water or liquid fertilizer as the carrier, and postemergence applications are to be made with crop oil concentrate (1 gal/100 gal water; 1.0% v:v). Use of methylated seed oil (MSO) or MSO blend adjuvants is prohibited, and UAN (28-0-0) and AMS additives are not to

be used with applications to sweet corn. Corn is not to be cultivated 7 days prior to or following application.

The following rotational crop restrictions are proposed for the 4 lb/gal FlC formulation: a plantback interval of 0 days for corn; a plantback interval of 120 days for small grains; and a plantback interval of 18 months for all other rotational crops other than soybean, sorghum, cotton, potato, sunflower, canola, tobacco, and sweet corn. Soybean, sorghum, cotton, potato, sunflower, canola, tobacco, and sweet corn may be replanted the following season.

We note that in the previous petition, the following label amendments were required: (i) proposal of a preharvest interval (PHI) for field corn forage; the available field trial data support a PHI of 45 days; and (ii) revision to specify a minimum spray volume of 10-30 gal *water*/A and 3 gal *water*/A when applying Callisto™ by ground or aerial application, respectively. It was recommended that the petitioner add a statement to the label specifying that postemergence applications must be made at the V3 to V8 leaf corn growth stage for field corn. It was noted that if it was the petitioner's intention not to exceed a maximum seasonal rate of 0.43 lb ai/A, the following combinations of two applications may be applied: two postemergence applications [maximum total = 0.188 lbs ai/A (0.094 lbs ai/A + 0.094 lbs ai/A)]; two preemergence applications [maximum total = 0.43 lbs ai/A (0.24 lbs ai/A + 0.188 lbs ai/A)]; and one preemergence and one postemergence applications [maximum total = 0.33 lbs ai/A (0.24 lbs ai/A + 0.094 lbs ai/A)].

Based on examination of the registered label for the 4 lb/gal FlC formulation (EPA Reg. No. 100-1131, dated 01/14/2004), the petitioner has modified the label: (i) to specify a 45-day PHI for field corn forage; (ii) to specify that preemergence ground applications be made in 10-80 gal/A using water or liquid fertilizer as the carrier; (iii) to specify that the product is not to be applied preemergence using aerial equipment; and (iv) to specify that field corn may be treated up to the 8-leaf growth stage, or when corn is up to 30 inches in height. A carrier for postemergence applications is specified in the specific crop directions (water containing crop oil concentrate). A maximum seasonal rate of 0.24 lb ai/A has been specified for preemergence applications, with a maximum of two applications per season; if only postemergence applications are made, the maximum seasonal rate is 0.19 lb ai/A.

We also note that the original field corn petition proposed registration of a suspension concentrate (FlC) formulation only; the field trial data submitted with that petition reflected application of the FlC formulation. Currently, three EC formulations of mesotrione are registered for use on field corn (EPA Reg. Nos. 100-1142, 100-1148, and 100-1152) in addition to the FlC formulation. One of the formulations, EPA Reg. No. 100-1142, is registered for preemergence use only, at a maximum of 0.24 lb ai/A. The other two formulations, EPA Reg. Nos. 100-1148 and 100-1152, are registered for preemergence use or early postemergence use (when plants are up to 5 inches in height) at a maximum of 0.20 lb ai/A. There are no available field corn field trial data reflecting application of an EC formulation. TRB allows translation of crop field trial data between formulations which are diluted with water and applied prior to crop emergence (i.e., preplant, at-plant, and preemergence applications) or just after crop emergence. TRB concludes that because application of the EC formulations to field corn is restricted to

preemergence and early postemergence use, crop field trial data for the FlC formulation may be translated to support use of the EC formulations.

Conclusions. The proposed use directions for the 4 lb/gal FlC formulation are adequate. The petitioner should correct the rotational crop restriction to indicate that field corn and sweet corn have a 0-day plantback interval and remove sweet corn from the list of crops which cannot be rotated until the following season.

Note to PM: The label for the 4 lb/gal FlC formulation indicates tank mix uses on sweet corn with the following products which do not currently have registered uses on sweet corn: Axiom®, Bicep®, Bicep Lite II®, Degree™, Degree Xtra™, Doubleplay®, Dual II®, Fultime™, Harness®, Harness Xtra®, Liberty®, Liberty ATZ, Surpass® EC, and Topnotch®. These tank mixes should be removed.

860.1300 Nature of the Residue - Plants

PP#8F04954, DP Barcodes: D245477 and D260267, 6/6/01, S. Levy
DP Barcode: D274111, 4/26/01, S. Levy

No new plant metabolism studies were submitted with the current petition. Field corn metabolism studies, reflecting labeling in the cyclohexane (CY) and phenyl (PH) rings of mesotrione, were previously submitted in conjunction with the petition (PP#8F04954, DP Barcodes: D245477 and D260267, 6/6/01, S. Levy) for field corn uses. It was concluded that, pending submission of additional storage stability data and information for the CY-label study, the qualitative nature of the residue in corn was adequately understood.

Total radioactive residues (TRR), following a single preemergence application of uniformly ring-labeled [PH-^{14}C]mesotrione or [CY-^{14}C]mesotrione at 0.250-0.273 lb ai/A (1.0-1.1x the maximum seasonal, preemergence, rate to field corn) were, respectively, 0.013 and 0.001 ppm in corn grain and 0.795 and 0.015 ppm in corn stover harvested at maturity (153 days after treatment, DAT). TRR were 0.356 and 0.067 ppm in immature corn forage harvested 27-28 days after the preemergence application of [PH-^{14}C]mesotrione or [CY-^{14}C]mesotrione, respectively. In a second plot of corn which received a single postemergence foliar application of [PH-^{14}C] or [CY-^{14}C]mesotrione at 0.144-0.146 lb ai/A (0.8x the maximum postemergence application rate), TRR were, respectively, 0.014 and 0.011 ppm in grain, 1.066 and 0.330 ppm in stover, and 0.244 and 0.098 ppm in immature forage harvested 28 DAT.

Mesotrione was identified at low levels in corn forage (0.4-3.0% TRR, 0.001-0.008 ppm) indicating that residues were extensively metabolized. Metabolite 4-OH ZA1296 was identified in forage and stover (0.7-3.8% TRR, 0.007-0.014 ppm) from the PH-label studies, and in forage from the CY-label studies at low levels (6.1-10.4% TRR, 0.006-0.007 ppm). Metabolites 4-OGlu ZA1296, MNBA [4-(methylsulfonyl)-2-nitrobenzoic acid] and AMBA [2-amino-4-(methylsulfonyl)benzoic acid] were only identified in corn matrices from the PH studies; AMBA (free, acid-labile, base-labile, and hexose esters) metabolite levels were greater following postemergence treatment than preemergence treatment. Metabolite 4-OGlu ZA1296 was identified in forage from the PH studies at low levels (3.6-3.8% TRR, 0.009-0.013 ppm).

AMBA was the major metabolite identified in forage (12.2-13.2% TRR, 0.032-0.043 ppm) and stover (13.6-28.2% TRR, 0.108-0.301 ppm) from the PH studies. MNBA was also identified in PH forage (3.4-19.7% TRR, 0.008-0.070 ppm) and stover (1.0-1.9% TRR, 0.008-0.019 ppm). A large amount of residues were characterized as minor compounds with diverse polarity. In the PH studies, extensive attempts (acid, base, and enzyme hydrolysis, acidic and basic TLC) were made to further characterize the unknown polar components. Based on mobility and responses to hydrolysis, the unknowns were considered to be similar to 4-OGlu ZA1296 and were likely polar conjugates, with endocons of neutral or amphoteric character. In the CY studies, the incorporation of radioactivity into carbohydrates, such as glucose, fructose, and malic acid, was demonstrated. The incorporation of radioactivity into lignin and cellulose was also characterized in postemergence stover.

The metabolite profiles differed significantly in the PH and CY studies. Little of the residue characterized from the PH studies resulted from the fixation of ^{14}C, while the identification of carbohydrates in the CY studies indicate that incorporation of ^{14}C into biomolecules is the major source of radioactive residues.

In an additional study reflecting preemergence plus postemergence applications, TRR following a single preemergence application at 0.27 lb ai/A and subsequent postemergence application at 0.16 lb ai/A (total application rate of 0.43 lb ai/A; 1.3x the maximum seasonal rate) of [PH-^{14}C]mesotrione were 0.03 ppm in corn grain and 0.57 ppm in corn stover harvested at maturity (91 DAT), and 0.27 ppm in immature forage harvested 48 DAT. Residues of the parent, mesotrione, were not detected in this metabolism study. Metabolite 4-OH ZA1296 was identified only in corn forage at low levels (5.4% TRR, 0.01 ppm). AMBA, MNBA, and their conjugates were identified in both forage (2.2-4.6% TRR, 0.01 ppm) and stover (1.0-2.3% TRR, 0.01 ppm). As was observed in the separate preemergence and postemergence studies, minor compounds represented the largest amount of the radioactivity; however, each of these components was individually present at <0.01 ppm.

In response to the requirements for storage stability information for the CY-label study, Syngenta Crop Protection has submitted (MRID 45651813) information pertaining to sample extraction and analysis dates and a discussion of storage stability for the corn metabolism study. The previous review had concluded that the petitioner should provide information pertaining to the storage conditions and intervals of samples and extracts following initial processing until completion of final analyses, as well as data demonstrating that the integrity of the metabolite profile did not change significantly during storage (if final analyses were completed more than 4-6 months following sample collection).

In their response, Syngenta stated that the original submission demonstrated that all initial analyses were conducted within approximately 6 months of harvest of the samples. Samples and extracts were stored at -20 °C throughout the study. Forage samples were extracted and initially analyzed within 3.5 months of collection, and the extracts were further analyzed for residue identification within 6 months of collection. Additional analyses were conducted on forage approximately 18 months after collection, using the initial extracts. It was stated that these additional analyses were conducted to confirm the identification of mesotrione and 4-

hydroxymesotrione and the incorporation of radioactivity into natural products. The petitioner noted that stability of residues of mesotrione and 4-hydroxymesotrione was demonstrated in the PH-label study.

Grain samples were extracted and analyzed within 6.5 months of collection. A fodder subsample was extracted 3 months after collection, and a second subsample was extracted 10 months after collection. The extraction profiles from the initial and final fodder extractions were found to be similar. The aqueous fractions were subjected to HPLC analyses, which showed comparable metabolic profiles: a large amount of highly polar components (unretained radioactivity) as well as multiple components at low concentrations.

Conclusions. HED concludes that the petitioner has adequately responded to the requirements for additional storage stability data for the CY-label field corn metabolism study.

The MARC (meeting of 4/22/97, memorandum dated 6/20/97, J. Stokes) had previously determined, from a preliminary briefing on the metabolism and field trial studies (memorandum dated 3/20/97, J. Stokes), that the registrant should analyze all field trial samples for mesotrione and MNBA, the major soil metabolite, in target and rotational crops; the Committee had concluded that MNBA, a precursor of the AMBA, could be used as a potential marker for residues of AMBA for risk assessments if necessary because of difficulties associated with analysis for AMBA. The MARC met again on 4/10/01 (DP Barcode: D274111, 4/26/01, S. Levy) to consider the mesotrione plant, animal, and rotational crop metabolism studies. The MARC concluded that for the tolerance expression and risk assessment purposes, the residue of concern in/on field corn commodities is mesotrione *per se*.

860.1300 Nature of the Residue - Livestock

PP#8F04954, DP Barcodes: D245477 and D260267, 6/6/01, S. Levy
DP Barcode: D274111, S. Levy and D. Nixon, 4/26/01

No new livestock metabolism studies were submitted with the current petition. Cattle and hen metabolism studies, reflecting labeling in the cyclohexane (CY) and phenyl (PH) rings of mesotrione, were previously submitted in conjunction with the petition (PP#8F04954, DP Barcodes: D245477 and D260267, 6/6/01, S. Levy) for field corn uses. It was concluded that, pending submission of additional storage stability data and information for the CY-label cattle study, the qualitative nature of the residue in livestock was adequately understood.

Cattle: Following oral administration of [PH-^{14}C]mesotrione to a dairy cow for seven consecutive days at a feeding level of 11.91 ppm (25.8x and 22.6x the maximum theoretical dietary burden to beef and dairy cattle, respectively; see Table 6), the TRR (expressed as mesotrione equivalents) were 0.01-0.03 ppm in milk, 0.077 ppm in liver, 0.067 ppm in kidney, 0.002 ppm in muscle, ≤0.004-0.007 ppm in fat. Residues in milk plateaued on Day 2, and residues in tissues were highest in liver.

Following oral administration of [CY-2-^{14}C]mesotrione to a dairy cow for seven consecutive days at a feeding level of 9.9 ppm (21.5x and 18.8x the maximum theoretical dietary burden to

beef and dairy cattle, respectively), the TRR (expressed as mesotrione equivalents) were 0.006-0.079 ppm in milk, 0.110 ppm in liver, 0.110 ppm in kidney, 0.007 ppm in muscle, and 0.005-0.013 ppm in fat. Residues in milk plateaued on Day 4-5, and residues in tissues were highest in liver and kidney.

The parent, mesotrione, was identified as the major metabolite in kidney (18.0% TRR, 0.012 ppm) and liver (10.3% TRR, 0.009 ppm) from the PH study. From the CY study, the parent, mesotrione, was also identified as the major metabolite in kidney (14.4% TRR, 0.015 ppm) and liver (12.5% TRR, 0.013 ppm). From the PH study, AMBA was only identified in kidney (15.0% TRR, 0.010 ppm). From the CY study, [^{14}C]lactose was identified as the major metabolite in milk (35.1% TRR, 0.025 ppm). Unknowns, none of which were present at >0.010 ppm, were characterized as aqueous or organosoluble. Additional characterization of the tissue extracts determined that a significant amount of radioactivity was associated with proteinaceous material in the liver.

In a separate study, following oral administration of [PH-^{14}C]AMBA to a dairy cow for seven consecutive days at a feeding level of ~10 ppm, the TRR (expressed as AMBA equivalents) were 0.0030-0.0090 ppm in milk, 0.005 ppm in liver, 0.053 ppm in kidney, 0.000 ppm in muscle, and 0.000-0.018 ppm in fat. Residues in milk plateaued on Day 3, and residues in tissues were highest in kidney.

AMBA was identified in kidney (79.0% TRR, 0.038 ppm) and perirenal fat (61.6% TRR, 0.013 ppm). The petitioner stated that since AMBA was the major residue identified in kidney and perirenal fat, and also in urine (95.4% TRR), residues in kidney and perirenal fat are most likely a result of residual urine remaining in these tissues.

In response to the requirements for storage stability information for the CY-label study, Syngenta Crop Protection has submitted (MRID 45651814) information pertaining to sample extraction and analysis dates and a discussion of storage stability for the cattle metabolism study. The previous review had concluded that the petitioner should provide information pertaining to the storage intervals of samples and extracts following initial processing until completion of final analyses, as well as data demonstrating that the integrity of the metabolite profile did not change significantly during storage (if final analyses were completed more than 4-6 months following sample collection).

In their response, Syngenta stated that the original submission demonstrated that all initial analyses were conducted within 6 months of collection of the samples. Samples and extracts were stored frozen throughout the study. Certain HPLC analyses of extracts were conducted later than 6 months after sample collection, to confirm the results of the initial TLC analyses.

Based on the dates provided in their response, initial extraction and TLC profiling of milk and liver samples was conducted within 48 days of collection, and final analyses were completed within 167 days (5.5 months) and 201 days (6.6 months) for milk and liver, respectively. Kidney samples were extracted and initial TLC profiles completed within 161 days (5.3 months) of

collection, and final analyses were completed within 208 days (6.8 months). Muscle and fat samples were not subjected to extraction and analysis, because of low radioactivity levels.

Hen: Following oral administration of [PH-^{14}C]mesotrione to laying hens for 10 consecutive days at a feeding level of ~10 ppm (~1250x the maximum theoretical dietary burden to poultry), the TRR (expressed as mesotrione equivalents) were <0.003 ppm in egg whites, <0.003-0.024 ppm in egg yolks, 1.121 ppm in liver, ≤0.004 ppm in muscle, <0.003 ppm in peritoneal fat, and 0.042 ppm in subcutaneous fat with skin. Residues in egg yolks plateaued on the sixth day of dosing. Residues in tissues were highest in liver.

Following oral administration of [CY-^{14}C]mesotrione to laying hens for 10 consecutive days at a feeding level of ~10 ppm (~1250x), the TRR (expressed as mesotrione equivalents) were 0.012-0.025 ppm in egg whites, 0.002-0.094 ppm in egg yolks, 1.245 ppm in liver, 0.011-0.012 ppm in muscle, 0.010 ppm in peritoneal fat, and 0.048 ppm in subcutaneous fat with skin. Residues in egg whites and yolks plateaued on the fifth and eighth days of dosing, respectively. Residues in tissues were highest in liver.

The parent, mesotrione was the only identified compound; it was found in egg yolks, subcutaneous fat, and liver at 24-91% TRR (0.017-1.097 ppm). Incorporation of radioactivity into naturally occurring fatty acids such as palmitic, oleic, and stearic acid was tentatively observed in CY egg yolk. Additional unknowns were observed in both PH and CY egg and tissue matrices; each unknown fraction accounted for <0.01 ppm.

Conclusions. TRB concludes that the petitioner has adequately responded to the requirements for additional storage stability data for the CY-label cattle metabolism study.

The MARC reviewed and discussed the nature of the residue in livestock commodities and concluded that for the tolerance expression and risk assessment purposes, the residue of concern in/on livestock commodities is mesotrione *per se* (Memo, S. Levy and D. Nixon, 4/26/01, DP Barcode: D274111). It was noted that if in the future the registrant proposes a use which increases the maximum theoretical dietary burden, the conclusion would be re-evaluated.

860.1340 Residue Analytical Methods

45651802.der.wpd
DP Barcode: D261112, J. Negron, 8/17/01
PP#8F04954, DP Barcodes: D245477 and D260267, 6/6/01, S. Levy

Plant commodity methods

Enforcement method: The current enforcement method for plant commodities is an HPLC method with fluorescence detection (FLD), Method TMR0882B. This method was reviewed in conjunction with the field corn petition (PP#8F04954, DP Barcodes: D245477 and D260267, 6/6/01, S. Levy) and has undergone adequate petition method validation (DP Barcode: D261112, J. Negron, 8/17/01) .

In the previous petition, HED had concluded that the petitioner must submit a new interference study for the enforcement method for plant commodities. Syngenta has responded to that requirement (MRID 45651816) by instead submitting a confirmatory method for mesotrione residues in corn commodities. Syngenta has proposed an LC/MS/MS method, RAM 366/01, for the confirmation of residues of mesotrione and MNBA in corn commodities. The method is entitled "Residue Analytical Method for the Determination of Residues of Mesotrione and 4-(Methylsulfonyl)-2-Nitrobenzoic Acid (MNBA) in Crop Samples."

Samples are extracted with acetonitrile/water, and an aliquot of the extract is cleaned up by solid phase extraction. Residues of mesotrione and MNBA are dissolved in water/methanol and analyzed by HPLC with MS/MS detection. The validated limit of quantitation (LOQ) is 0.01 ppm for each analyte in corn commodities. The limits of detection (LODs) were reported to be 0.002 ppm for mesotrione and 0.005 ppm for MNBA.

Method validation data for LC/MS/MS method RAM 366/01 demonstrated adequate recoveries of mesotrione and MNBA at the LOQ, 10x the LOQ, and 100x the LOQ from corn whole plant and seed, and at 1000x the LOQ from corn whole plant. Adequate independent laboratory validation data have been submitted for corn grain, stover, and silage at 1x, 2x, and 10x the LOQ.

Data collection method: Samples of sweet corn commodities from the submitted sweet corn field trials were analyzed for residues of mesotrione and its metabolite MNBA using Method TMR0882B, the current enforcement method for field corn commodities. The validated LOQ was 0.01 ppm for each metabolite in sweet corn forage, ears, and stover. This method is adequate for data collection based on acceptable concurrent method recovery data.

Conclusions. The submitted LC/MS/MS method, method RAM 366/01, is adequate for use as a confirmatory method for enforcement purposes. The method will be forwarded to FDA for inclusion in PAM Volume II as a confirmatory method. Validation by EPA/ACB is not required for this method.

The current enforcement method for field corn commodities, HPLC/FLD method TMR0882B, is adequate for use as an enforcement method for sweet corn commodities. Additional validation by EPA/ACB is not required.

Livestock commodity methods

Because no tolerances have been established or proposed for livestock commodities in conjunction with the registered and proposed uses of mesotrione, no livestock commodity methods have been submitted.

860.1360 Multiresidue Methods

The petitioner submitted multiresidue method data with the previous petition, which were forwarded to FDA for full evaluation. The FDA PESTDATA database dated 11/01 (PAM Volume I, Appendix I) indicates that mesotrione is not recovered using Multiresidue Methods Sections 302 (Luke Method; Protocol D). No recovery data pertaining to Multiresidue Methods Section 303 (Mills, Onley, and Gaither Method; Protocol E, nonfatty food) or 304 (Mills Method; Protocol F, fatty food) were included.

860.1380 Storage Stability

Storage stability data, submitted with the previous petition (PP#8F04954, DP Barcodes: D245477 and D260267, 6/6/01, S. Levy) for field corn uses, demonstrated that residues of mesotrione and its metabolite MNBA were relatively stable in/on various raw agricultural commodities stored under frozen conditions. Fortified residues of mesotrione and its metabolite MNBA were stable for up to 42 months in/on field corn forage, fodder, and grain, up to 44 months in/on radish root, and up to 40 months in/on soybean seed.

The maximum storage intervals of sweet corn samples from the submitted sweet corn field trials were 220 days (7.2 months) for forage, 209 days (6.9 months) for ears, and 169 days (5.6 months) for stover.

Conclusions. The available storage stability data for field corn commodities are adequate to support the storage intervals and conditions of samples of sweet corn commodities from the submitted sweet corn field trial data.

860.1480 Meat, Milk, Poultry, and Eggs

No livestock feeding study data were submitted with this petition. For the field corn petition, HED had concluded that there was no reasonable expectation of quantifiable mesotrione residues of concern in eggs, milk, and the meat, fat, or meat byproducts of poultry and ruminants as a result of the proposed uses [Category 180.6(a)(3)].

The maximum theoretical dietary burden to livestock based on field corn uses was calculated to be 0.0182 ppm to cattle and 0.01 ppm to poultry. The proposed uses on sweet corn result in a significantly higher dietary burden to cattle. The maximum theoretical dietary burden of mesotrione to livestock based on the proposed and registered uses is presented in Table 6.

Table 6. Calculation of Maximum Dietary Burden of Mesotrione to Livestock.

Feedstuff	% Dry Matter[1]	% Diet[1]	Estimated Tolerance (ppm)	Dietary Contribution (ppm)[2]
Beef Cattle				
Sweet corn stover	83	25	1.5	0.452
Field corn grain	88	75	0.01	0.009
TOTAL BURDEN	--	100	--	**0.461**

Table 6. Calculation of Maximum Dietary Burden of Mesotrione to Livestock.				
Feedstuff	% Dry Matter[1]	% Diet[1]	Estimated Tolerance (ppm)	Dietary Contribution (ppm)[2]
Dairy Cattle				
Sweet corn forage	48	50	0.50	0.521
Field corn grain	88	40	0.01	0.005
TOTAL BURDEN	--	90 [3]	--	**0.526**
Poultry				
Field corn grain	--	80	0.01	0.008
TOTAL BURDEN	--	80 [3]		**0.0080**
Swine				
Field corn grain	--	80	0.01	0.008
TOTAL BURDEN	--	80 [3]		**0.0080**

[1] Table 1 (*OPPTS Guideline 860.1000*).

[2] Contribution = ([tolerance /% DM] x % diet) for beef and dairy cattle; Contribution = (tolerance x % diet) for poultry and swine.

[3] The remainder of the diet will be composed of feedstuff derived from crops that do not have existing or proposed mesotrione uses.

The maximum residues of mesotrione observed in the cattle metabolism studies were 0.012 ppm and 0.015 ppm, in kidney from the PH-labeled feeding level of 11.91 ppm and the CY-labeled feeding level of 9.90 ppm. Based on these dosing levels, which are 22.6x and 18.8x the beef cattle dietary burden from Table 6, above, the maximum expected residues of mesotrione in cattle commodities would be 0.0005 and 0.0008 ppm.

The petitioner has submitted (MRID 45651817) a discussion of the need for livestock feeding studies. The petitioner noted that sweet corn commodities are not significant poultry feedstuffs. Therefore, there is no change in the theoretical dietary burden to poultry based on the proposed uses on sweet corn.

The petitioner calculated a theoretical dietary burden to beef cattle of 1.02 ppm based on a diet consisting of 35% field corn grain, 25% sweet corn stover, and 40% sweet corn forage. The petitioner also calculated transfer factors from the average results of the two cattle metabolism studies; transfer factors were calculated by dividing the recovered ^{14}C-mesotrione residues by the total amount of ^{14}C-mesotrione dosed. Because of low residues in muscle and fat, transfer factors were only calculated for kidney and liver (0.0013 and 0.0010, respectively). Based on the theoretical diet and the transfer factors, the petitioner calculated that expected mesotrione residues in beef kidney and liver would be 0.0013 and 0.0010 ppm and concluded that cattle feeding study data are not required.

Conclusions. Based on the cattle metabolism studies dosing levels, which are 22.6x and 18.8x the beef cattle dietary burden from Table 6, above, the maximum expected residues of mesotrione in cattle commodities would be 0.0005 and 0.0008 ppm. In consideration of these values, the length of duration of dosing in the cattle metabolism studies (7 days), and that residues in milk had plateaued by the second to fifth day of dosing in the metabolism studies,

TRB concludes that there is no expectation of finite residues in livestock commodities for the proposed uses on sweet corn. Should a future proposed use significantly increase the calculated burden, a cattle feeding study may be required.

Because the proposed uses on sweet corn do not increase the dietary burden to poultry, HED concludes that no poultry feeding study is required. There is no expectation of finite residues in poultry commodities.

860.1500 Crop Field Trials

45651801.der.wpd (Sweet corn)

Table 7. Summary of Residues from the Crop Field Trials with Mesotrione.

Crop Matrix	Total Applic. Rate (lb ai/A)	PHI (days)	Analyte	Residue Levels (ppm) [1]					
				n	Min.	Max.	HAFT	Mean	Std. Dev.
Sweet corn (proposed use = 0.34 lb ai/A total application rate, 45-day PHI)									
Forage, with ears	0.428-0.451	14	Mesotrione	24	<0.01	0.879	0.807	0.079	0.226
			MNBA	24	<0.01	0.061	0.054	0.010	0.014
Forage, without ears		26-31	Mesotrione	24	<0.01	0.396	0.389	0.039	0.108
			MNBA	24	<0.01	0.037	0.034	0.009	0.010
Ears		26-31	Mesotrione	24	<0.01	<0.01	<0.01	0.005	0.0
			MNBA	24	<0.01	<0.01	<0.01	0.005	0.0
Stover		52-71	Mesotrione	24	<0.01	1.204	1.077	0.094	0.305
			MNBA	24	<0.01	0.075	0.071	0.010	0.019

[1] For the determination of minimum, maximum, and HAFT values, the LOQ (<0.01 ppm) was used for residues reported as <LOQ. For the determination of the median, mean, and standard deviation values, half the LOQ (0.005 ppm) was used for residues reported as <LOQ. HAFT = Highest Average Field Trial.

Sweet corn: Syngenta Crop Protection, Inc. has submitted crop field trial data for sweet corn. A total of twelve sweet corn field trials were conducted during the 2001 growing season in Regions I (NY and PA), II (NC), III (FL), V (IL, MI, MN, OH, and WI), X (CA), XI (ID), and XII (WA). The number and locations of field trials are in accordance with OPPTS Guideline 860.1500 for sweet corn.

A single preemergence application of the 4 lb/gal FlC formulation was made at ~0.270 lb ai/A to the soil surface preplant (on the day of planting) or within 2 days after planting of sweet corn (1.1x the maximum proposed preemergence application rate), followed, 43-116 days later, by a single postemergence foliar application of the 4 lb/gal FlC formulation at ~0.16 lb ai/A (1.7x the maximum proposed single postemergence application rate). Total application rates (pre- and postemergence) ranged 0.428-0.451 lb ai/A (1.3x the proposed maximum seasonal rate). Sweet corn forage (with ears) was harvested 14 days posttreatment, forage (without ears) and ears were harvested 26-31 days posttreatment, and stover was harvested 52-71 days posttreatment. Additional samples of forage (with and without ears) and ears were collected from two field trials to evaluate residue decline. The petitioner stated (in the summary of the field trial procedures)

that adjuvants [SOC or COC at 1% (v:v) and AMS or UAN at 2.5% (v:v)] were added to the spray mixtures for postemergence applications. However, no information pertaining to spray adjuvants was included in the field trial summaries (field trial raw data).

Samples of sweet corn commodities were analyzed for residues of mesotrione and its metabolite MNBA using an HPLC method with fluorescence detection, Method TMR0882B, the current enforcement method for field corn commodities. The validated LOQ was 0.01 ppm for each metabolite in sweet corn forage, ears, and stover. This method is adequate for data collection based on acceptable concurrent method recovery data.

Residues of mesotrione were <0.01-0.879 ppm in/on sweet corn forage (with ears) harvested 14 days posttreatment, <0.01-0.396 ppm in/on sweet corn forage (without ears) harvested 26-31 days posttreatment, <0.01 ppm in/on sweet corn ears harvested 26-31 days posttreatment, and <0.01-1.204 ppm in/on sweet corn stover harvested 52-71 days posttreatment. Residues of MNBA were <0.01-0.061 ppm in/on sweet corn forage (with ears), <0.01-0.037 ppm in/on sweet corn forage (without ears), <0.01 ppm in/on sweet corn ears, and <0.01-0.075 ppm in/on sweet corn stover.

We note that residues of mesotrione were at or below the LOQ (≤0.01 ppm) in/on samples of forage (without ears) collected 26-31 days posttreatment from all field trials except two (CA and ID), in/on ear samples from all field trials, and in/on stover samples from all field trials except one (CA). In the ID field trial, residues of mesotrione were <0.01 and 0.052 ppm in/on forage (without ears) harvested 30 days posttreatment. In the CA field trial, residues of mesotrione were much higher than in the other trials; residues were 0.381 and 0.396 ppm in/on forage (without ears) harvested 30 days posttreatment, and residues were 0.949 and 1.204 ppm in/on stover harvested 58 days posttreatment. Residues of mesotrione were quantifiable, ranging 0.017-0.879 ppm, in/on forage (with ears) samples harvested 14 days posttreatment in four of the field trials, and nonquantifiable (<0.01 ppm) in/on all forage (with ears) samples from the other eight field trials.

Data from the residue decline studies demonstrate that residues of mesotrione and MNBA decline in forage (with and without ears) with increasing harvest intervals. In one residue decline trial (CA), average residues of mesotrione and MNBA in/on forage (with ears) declined from 4.63 and 0.093 ppm, respectively, at the 0-day sampling interval, to 0.807 and 0.054 ppm, respectively, at the 14-day sampling interval. Average residues of mesotrione and MNBA in/on forage (without ears) declined from 0.398 and 0.039 ppm, respectively, at the 23-day sampling interval, to 0.281 and 0.030 ppm, respectively, at the 37-day sampling interval. In the second residue decline trial (MI), average residues of mesotrione and MNBA in/on forage (with ears) declined from 3.53 and 0.048 ppm, respectively, at the 0-day sampling interval, to <0.01 ppm (both analytes) at the 14-day sampling interval; residues in/on forage (without ears) were below the LOQ in/on all samples from this decline study. Residues in sweet corn ears were below the LOQ in all samples from both decline studies.

The submitted sweet corn field trial residue data were submitted with conflicting information as to the use of spray adjuvants. The summary indicated that spray adjuvants were used while the

raw field trial data did not indicate whether they were used or not (MRID: 45651801). The registrant submitted additional information describing the spray adjuvants used at each field trial site for postemergence applications, including the concentration of each adjuvant added to the spray mixture (Attachment 1). The use of spray adjuvants is indicated by the **Directions for Use**, above and was used for the crop field trials.

Conclusions: The submitted sweet corn field trial data support the following tolerances for residues of mesotrione *per se*: sweet corn kernel plus cob with husks removed at 0.01 ppm and sweet corn forage at 0.50 ppm. The requested tolerance on sweet corn stover at 2.0 ppm is too high; a tolerance of 1.5 ppm is more appropriate. A revised Section F is required.

860.1520 Processed Food and Feed

HED does not require residue data for any processed commodities of sweet corn. Adequate field corn processing data were submitted under the previous petition (PP#8F04954, DP Barcodes: D245477 and D260267, 6/6/01, S. Levy) for field corn uses which indicated that tolerances for mesotrione residues were not required for field corn processed commodities.

860.1650 Submittal of Analytical Reference Standards

DP Barcode:D261112, J. Negron, 8/17/01

The analytical reference standards for mesotrione have been submitted to the EPA National Pesticide Standards Repository.

860.1850 Confined Accumulation in Rotational Crops

DP Barcode: D274111, 4/26/01, S. Levy
PP#8F04954, DP Barcodes: D245477 and D260267, 6/6/01, S. Levy

No new confined rotational crop studies were submitted with this petition. An adequate confined rotational crop study was submitted under the previous petition (PP#8F04954, DP Barcodes: D245477 and D260267, 6/6/01, S. Levy) for field corn uses. Total radioactive residues (TRR) expressed as [^{14}C]mesotrione equivalents, accumulated at levels ≥0.01 ppm in the following rotational crop commodities of soybeans and wheat planted in sandy loam soil 30 days after treatment (DAT) with uniformly ring-labeled phenyl (PH) or cyclohexane-labeled (CY) [^{14}C]mesotrione at 0.274 lb ai/A (1.1x the maximum proposed/registered preemergence application rate for corn): soybean forage, hay, and soybeans, and wheat forage, hay, straw, and grain. TRR in PH samples ranged from 0.038 ppm in wheat grain to 2.58 ppm in wheat straw; in CY samples TRR were lower, ranging from 0.010 ppm in wheat grain to 0.059 ppm in wheat straw.

In rotational crop commodities of endive, radish, and wheat planted 120 days and 300 days after treatment of the soil with PH or CY [^{14}C]mesotrione at 0.411 lb ai/A (1.2x the maximum proposed seasonal rate for sweet corn), TRR accumulated at levels ≥0.01 ppm in 120-DAT PH endive, radish tops and roots, and wheat forage, hay, 120-DAT CY wheat forage, hay, and straw,

and 300-DAT PH endive, radish tops, and wheat forage, hay, straw, and grain. TRR in 120-DAT PH samples ranged from 0.014 ppm in wheat grain to 0.303 ppm in wheat forage; in 120-DAT CY samples, TRR ranged from 0.13 ppm in wheat hay to 0.043 ppm in wheat straw. TRR in 300-DAT PH samples ranged from 0.015 ppm in wheat grain to 0.197 ppm in wheat straw.

Approximately 51-97% TRR were identified/characterized in rotational crop commodities. Mesotrione was identified at <0.01 ppm in rotational crop commodities, accounting for 1% and 2% TRR in 30-DAT PH wheat forage and 300-DAT PH wheat hay, and for 6-11% TRR in 30-DAT CY soybean and wheat commodities and 5% and 3% TRR in 120-DAT CY wheat forage and straw. MNBA and AMBA (free and conjugated) were the major identified metabolites in PH samples, but were not identified in CY samples. MNBA was identified at 8-62% TRR (0.003-0.625 ppm) in all samples analyzed from each rotational interval. Free AMBA accounted for 9-17% TRR (0.024-0.330 ppm) in 30-DAT samples except wheat grain, 5-16% TRR (0.012-0.020 ppm) in120-DAT wheat forage, hay, and straw, and 6-7% TRR (0.001-0.014 ppm) in all 300-DAT samples analyzed. AMBA sulfate conjugate was a major metabolite in 30-DAT (9-17% TRR, 0.088-0.317 ppm), 120-DAT (8-12% TRR, 0.015-0.028 ppm), and 300-DAT (11-21% TRR, 0.009-0.026 ppm) wheat forage, hay, and straw, and was identified in 120-DAT radish roots at 8% TRR (0.003 ppm). Another AMBA conjugate was identified at 1-7% TRR (0.001-0.021 ppm) in wheat forage, hay, and straw from all rotations. In PH samples of 30-DAT soybeans and wheat grain, glucose accounted for 3% and 34% TRR (0.004 ppm and 0.013 ppm). Residues of the following metabolites were identified in rotational crops at 1-15% TRR, (<0.001-0.014 ppm): 4-OH ZA1296, 5-OH ZA1296 (CY only), 4-O-glucose ZA1296 (CY only), and tetrahydro xanthenone ZA1296 (CY only).

As in the field corn metabolism study, the majority of residues characterized/identified in CY rotational crop commodities resulted from incorporation of $^{14}CO_2$ into natural products. Radioactivity was mostly unretained in the extractable fractions, and was characterized as carbohydrates such as glucose by HPLC analysis.

Conclusions. The available data are adequate to satisfy the requirements for confined rotational crop data for the purposes of this petition. Based on the components identified, the results of the confined rotational crop study suggest that mesotrione is metabolized in rotational crops via a route similar to that demonstrated in primary crops. The MARC (meeting of 4/22/97, memorandum dated 6/20/97, J. Stokes) had previously determined, from a preliminary briefing on the metabolism and field trial studies (memorandum dated 3/20/97, J. Stokes), that the registrant should analyze all field trial samples for mesotrione and MNBA, the major soil metabolite, in target and rotational crops; the Committee had concluded that MNBA, a precursor of the AMBA, could be used as a potential marker for residues of AMBA for risk assessments if necessary because of difficulties associated with analysis for AMBA. The MARC met again on 4/10/01 to consider the mesotrione plant, animal, and rotational crop metabolism studies (DP Barcode: D274111, 4/26/01, S. Levy). The Committee concluded that for the tolerance expression and risk assessment purposes, the residue of concern in/on rotational crop commodities is mesotrione *per se*.

860.1900 Field Accumulation in Rotational Crops

No new field rotational crop data were submitted with this petition. An acceptable limited field rotational crop study was submitted under the previous petition (PP#8F04954, DP Barcodes: D245477 and D260267, 6/6/01, S. Levy) for field corn uses. Residues of mesotrione and its metabolite MNBA were each less than the method LOQ (<0.01 ppm) in/on all rotational crop matrices (radish roots and tops; soybean forage, hay, and seed; millet forage, hay, straw, and grain; and sorghum forage) from the 29- to 30-day plantback interval (PBI) following a single preplant incorporated application made to the primary crop, field corn, of the 4 lb/gal FlC formulation at 0.30 lb ai/A/application (~0.9x maximum proposed seasonal rate to sweet corn). Residues of mesotrione and its metabolite MNBA were each less than the method LOQ (<0.01 ppm) in/on all rotational crop matrices (radish roots and tops; endive leaves; and wheat forage, hay, straw, and grain) from the 74- to 100-day PBI following two applications (preplant incorporated and postemergence) made to the primary crop, field corn, of the 4 lb/gal FlC formulation at a total rate of 0.50 lb ai/A (~1.5x maximum proposed seasonal rate).

Conclusions. The available field rotational crop data indicate that rotational crop tolerances will not be needed to support the proposed and registered uses on field and sweet corn, provided that a plantback interval of a minimum of 30 days is established for root crops (including bulb vegetables), soybeans, and small grains, and that a plantback interval of a minimum of 100 days is proposed for all other crops.

The following rotational crop restrictions are proposed for the 4 lb/gal FlC formulation: a plantback interval of 0 days for corn; a plantback interval of 120 days for small grains; and a plantback interval of 18 months for all other rotational crops other than soybean, sorghum, cotton, potato, sunflower, canola, tobacco, and sweet corn. Soybean, sorghum, cotton, potato, sunflower, canola, tobacco, and sweet corn may be replanted the following season. The proposed label states that crop injury may result if crops are planted at shorter plantback intervals than stated. Provided that the petitioner complies with the label amendments required under 860.1200, the proposed rotational crop restrictions are adequate.

860.1550 Proposed Tolerances

Syngenta Crop Protection, Inc. has proposed the establishment of permanent tolerances for residues of mesotrione in/on sweet corn commodities.

There are currently no established Codex, Canadian, or Mexican MRLs for mesotrione. An International Residue Limit Status sheet is attached to this review.

The proposed tolerances should be revised to reflect the correct commodity definitions as specified in Table 8.

Table 8. Tolerance Summary for Mesotrione.			
Commodity	Proposed Tolerance, ppm	Recommended Tolerance, ppm	Comments/ *Correct commodity definition*
Sweet corn grain (K+CWHR)	0.01	0.01	*Corn, sweet, kernel plus cob with husks removed*
Sweet corn forage	0.50	0.50	*Corn, sweet, forage*
Sweet corn stover	2.0	1.5	*Corn, sweet, stover*

Attachment 1. List of Spray Adjuvant Use
Attachment 2. International Residue Limit Status Sheet

Template Version November 2003

Attachment 1

Syngenta Crop Protection, Inc. HAES Dietary Safety Department

STUDY NUMBER: 487-01

TITLE: Mesotrione - Magnitude of the Residues in or on Sweet Corn

OBJECTIVE(S): To obtain data concerning the magnitude of the residues of mesotrione in or on sweet corn.

JUSTIFICATION: Data will be obtained in accordance with EPA Residue Chemistry Test Guidelines OPPTS 860.1000, "Background", and OPPTS 860.1500, "Crop Field Trials" (August 1996).

TEST
SUBSTANCE(S): See **SECTION I. A.** FIELD PHASE. TEST SUBSTANCE(S)

TEST SYSTEM: Sweet corn. (Variety/cultivar, source, and lot number will be added by protocol amendment to this study.)

I. D. TREATMENT METHODS, TIMING, AND EQUIPMENT

Test Substance Application: Application rates of the active ingredients, application method, frequency, interval, and spray volumes, if applicable, are outlined below. Refer to **I. C.** TEST LOCATIONS, **I. D.** TREATMENT METHODS, TIMING, AND EQUIPMENT, and the field-trial-specific Field Application and Sampling Information form(s) attached to this protocol.

Prepare enough test substance mixture to spray the entire treatment. Then divide the treatments into Rep A and Rep B.

Trt ID	Appl No	Application Formulation	Target App Rate	Application Timing	Method	GPA Min- Max	
1		Control					
2	1	Mesotrione 4SC	123 grams a.i./acre	Soil surface spray at planting	Broadcast Spray	10	20
	2	Mesotrione 4SC + SOC or COC at 1% v/v + AMS or UAN at 2.5 v/v	73 grams a.i./acre	30 days prior to harvest	Post Broadcast Spray*	10	20

For test substance calculations:

- Mesotrione 4SC (Callisto) contains 4 lbs. a.i./gallon of product.

*Make the post broadcast spray application 30 day prior to harvest. Add Superior Oil Concentrate (SOC) or crop oil concentrate (COC) at 1% v/v + ammonium sulfate (AMS) or urea-ammonium (UAN) at 2.5% v/v .

Excerpts from individual electronic Field Trial Notebook (eFTN) Trial Notes that substantiates inclusion of adjuvant with application of the test substance:

EC-HR-002 -01, Region 1
Crop Management Strategies, Inc.
Germansville, PA
Daniel R. Ramsdell, Field Investigator

> *** dramsdell - 7/10/01 12:54:00 PM ***
> Application # 2 spray mixture included Booster Plus E (COC) and UAN at 1% and 2.5% v/v respectively. The actual mix was as follows: 20 ml of COC, 50 ml of UAN, 1930 ml of water, and 4.4 ml of test substance (mesotrione 4SC).

EE-HR-003-01, Region 1
A.C.D.S. Research, Inc.
North Rose, NY
Grant L. Jordan, Principal Field Investigator

> *** glj - 07/16/2001 9:41:00 AM ***
> For the 2nd application, 26 ml of Crop Oil Concentrate and 66 ml of UAN (32% Nitrogen) were included in the spray mix. Spraye Mix = 2540.2 ml Water + 5.8 ml Mesotrione 4SC + 26 ml COC + 66 ml UAN = 2638 ml.

SJ-HR-003-01, Region 2
Agricultural Systems Associates
Rose Hill, NC
Paul Garvey, Senior Research Biologist

> *** Paul Garvey - 7/12/2001 12:35:00 PM ***
> Spray carrier solution included 2.5% v/v (75 ml) of AMS Plus and 1% (30ml) of crop oil concentrate.

VB -HR-101 -01, Region 3
Syngenta Vero Beach Research Center
Vero Beach, FL
Eric Rawls, PFI,Scientist

> *** Eric Rawls - 10/19/01 9:42:00 AM ***
> COC (Agridex FL-010718) was added to trt 2 at 1% v/v or 20 mls in the 2000 ml spray mixture. AMS FL-010546 was also added at 2.5% v/v or 50 grams in the 2000 ml spray mixture.

ED-HR-006 -01, Region 5
AGSEARCH COMPANY
Conklin, Michigan
Mark A. Waldecker, President AGSEARCH
Dan Platte, Research Assistant

*** Dan Platte - 8/13/01 8:53:00 AM ***
8/13/01 DP: Calibrate sprayer and made application #2.

Rigo Oil Concentrate (COC) @ 1% v/v = 47 ml

28% UAN @ 2.5% v/v = 118 ml

Carrier volume:4731 ml total - 12.8 ml Mesotrione - 47 ml COC - 118 ml UAN = 4553.2 ml water

N4-HR-003-01, Region 5
Syngenta Northern Regional Technical Center
Champaign, IL
Don C. Pullins, Technical Center Manager

*** D Pullins - 7/6/01 6:26:00 PM ***
Per protocol, 1% COC (40 mls) and 2.5% UAN (100 mls) were added to the spray mix. The addition of the adjuvants meant that only 3850 mls of carrier were required to make a 4000 ml mix.

NF-HR-002-01, Region 5
Warneke Research Services, LLC
Geneva, MN
Jon Warnke, PI

*** admn - 7/19/01 4:54:00 PM ***
Crop oil plus (COC) was added to the spray solution at a rate 1% (20 mls) along with UAN at a rate of 2.5% (50 mls).

NI-HR-001-01, Region 5
Great Lakes Ag-Research Service, Inc.
Delavan, WI
Susan K. Bellman, Principal Investigator
(Please note: Data was transcribed into eFTN. Adjuvant noted in archived raw data; however, not available electronically.)

*** Sue - 8/22/2001 9:19:00 AM ***
7/21/01 Received Soil Characterization Report back from Agvise.
SLF

8/7/01 Made the 2nd application today. Everything went well.
SLF

8/21/01 Transcribed 2nd application information into Advantage today.
Same comments concerning Advantage as for 1st applicaton. SKB

8/21/01 Form 13 of 2nd application on 8/7/01: Boom height was at the level of the tops of the tassels.

SKB

NK-HR-001 -01, Region 5
Ag Consultants, Inc.
New Holland, OH
John P. Gruber, President/General Manager
Carolyn Bonham, Office Manager/Archivist

*** cab - 8/2/2001 10:09:00 AM ***
The Actual Amount for Total Mixture Volume does not include crop oil or 28%.

W2-HR-102 -01, Region 10
Syngenta Western Regional Technical Center
Visalia, CA
Mike Ensminger, R & D Scientist III
Documentation of adjuvant included with application of test substance not available electronically.

WG-HR-005 -01, Region 11
AgraServe
Greenleaf, ID
Charles E. Osgood, Principal Field Investigator

*** ceosgood - 1/18/02 4:52:00 PM ***
Actual spray mix contained Moract COC at10.43 ml and UAN at 26.08 ml as per protocol.

WF-HR-003 -01, Region 12
Ron Britt & Associates, Inc.
Mt. Vernon, WA
Ron Britt, Research Director

*** Ron Britt - 12/28/01 11:43:00 AM ***
Application 2, 04 Sept. 2001, calibrated at 19.91 gals. per acre. total mix =
4000 mls.
3952 mls. water
40 mls. Omni Supreme Oil
8 mls. Mesotrione 4SC
12 grms. Ammonium sulfate

Attachment 2

INTERNATIONAL RESIDUE LIMIT STATUS

Chemical Name: 2-[4-(methylsulfonyl)-2-nitrobenzoyl]-1,3-cyclohexanedione	Common Name: Mesotrione	X Proposed tolerance ☐ Reevaluated tolerance ☐ Other	Date: 06/17/04

Codex Status (Maximum Residue Limits)	U. S. Tolerances
X No Codex proposal step 6 or above ☐ No Codex proposal step 6 or above for the crops requested	*Petition Number:* PP#2F6443 DP Barcode: D283827 Other Identifier:
Residue definition (step 8/CXL): N/A	Reviewer/Branch: Sarah Levy, RAB1
	Residue definition: Mesotrione

Crop (s)	MRL (mg/kg)	Crop(s)	Tolerance (ppm)
		Sweet corn grain (K+CWHR)	0.01
		Sweet corn forage	0.50
		Sweet corn stover	2.0

Limits for Canada	Limits for Mexico
X No Limits ☐ No Limits for the crops requested	X No Limits ☐ No Limits for the crops requested
Residue definition: N/A	Residue definition: N/A

Crop(s)	MRL (mg/kg)	Crop(s)	MRL (mg/kg)

Notes/Special Instructions: S. Funk, 07/12/04.

Rev. 1998

Mesotrione/122990/Syngenta Crop Protection
DACO 7.2.1, 7.2.2, 7.2.3 and 7.4/OPPTS 860.1340/OECD IIA 4.2.5, 4.2.6 and 4.3
Residue Analytical Method - Plant Commodities

Primary Evaluator	Dynamac Corp	Date: 1/12/05
Approved by	William Cutchin, Chemist TRB/RD	

This DER was originally prepared under contract by Dynamac Corporation. The DER has been reviewed by the TRB and revised to reflect current OPP policies.

STUDY REPORTS:

45651802 Bruns, G.; McLean, N; Nelson, S. (2001) Independent Laboratory Validation of the Analytical Method, "Residue Analytical Method for the Determination of Residues of Mesotrione and 4-(Methylsulfonyl)-2-Nitrobenzoic Acid (MNBA) in Crop Samples": Laboratory Study Number: 01SYN83.REP; Syngenta No. 2702-01. Unpublished study prepared by Syngenta Crop Protection, Inc. 92 p.

45651803 Crook, S.J. (2001) Residue Analytical Method for the Determination of Residues of Mesotrione and 4-(Methylsulfonyl)-2-Nitrobenzoic Acid (MNBA) in Crop Samples: RAM 366/01; Syngenta No. 2704-01. Unpublished study prepared by Syngenta Crop Protection, Inc. 45 p.

45651816 Williams, R. (2002) Syngenta Response to the Requirement of a Revised Interference Study as a Condition of Callisto Registration: Syngenta No. 1361-02. Unpublished study prepared by Syngenta Crop Protection, Inc. 5 p.

45665901 Hill, S.E. (2001) Validation of a Residue Analytical Method for the Determination of the Residues in Maize: Laboratory Study Number: RJ3253B; Syngenta No. 2701-01. Unpublished study prepared by Syngenta Crop Protection, Inc. 67 p.

EXECUTIVE SUMMARY:

In a previous petition (PP#8F04954; DP Barcodes D245477 and D260267, 6/6/01, S. Levy), Syngenta Crop Protection, Inc. had proposed an HPLC method with fluorescence detection for the enforcement of tolerances for mesotrione residues in plant commodities. The method (TMR0882B) has been accepted as an enforcement method; however, in the review of the petition, HED had concluded that the petitioner must submit a new interference study. Syngenta has responded to that requirement by instead submitting a confirmatory method for mesotrione residues in corn commodities.

Syngenta has proposed an LC/MS/MS method, RAM 366/01, for the confirmation of residues of mesotrione and MNBA in corn commodities. The method is entitled "Residue Analytical Method for the Determination of Residues of Mesotrione and 4-(Methylsulfonyl)-2-Nitrobenzoic Acid (MNBA) in Crop Samples."

Briefly, samples are extracted with acetonitrile/water, and an aliquot of the extract is cleaned up by solid phase extraction (SPE). Residues of mesotrione and MNBA are dissolved in water/methanol and analyzed by HPLC with MS/MS detection. The validated limit of quantitation (LOQ) is 0.01 ppm for each analyte in corn commodities. The limit of detection (LOD) was reported to be 0.002 ppm for mesotrione and 0.005 ppm for MNBA.

Method validation data for LC/MS/MS method RAM 366/01 demonstrated adequate recoveries of mesotrione and MNBA at the LOQ, 10x the LOQ, and 100x the LOQ from corn whole plant and seed, and at 1000x the LOQ from corn whole plant. Adequate independent laboratory validation data have been submitted for corn grain, stover, and silage at 1x, 2x, and 10x the LOQ.

STUDY/WAIVER ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS:

Under the conditions and parameters used in the study, the analytical method residue data are classified as scientifically acceptable. The method will be forwarded to FDA for inclusion in PAM Volume II as a confirmatory method. Validation by EPA/ACB is not required for this method.

The acceptability of this study for regulatory purposes is addressed in the forthcoming U.S. EPA Residue Chemistry Summary Document, DP Barcode D283827.

COMPLIANCE:

Signed and dated GLP, Quality Assurance and Data Confidentiality statements were provided. No deviations from regulatory requirements were reported.

A. BACKGROUND INFORMATION

Mesotrione is a triketone herbicide which inhibits the enzyme p-hydroxyphenylpyruvate dioxygenase (HPPD), disrupting carotenoid biosynthesis. This process leads to the destruction of chlorophyll, resulting in a bleaching effect in susceptible plants. Mesotrione is intended for preemergence and postemergence use for selective control of annual broadleaf weeds.

Mesotrione is currently registered for use on field and pop corn. The petitioner is now proposing uses on sweet corn.

TABLE A.1. Test Compound Nomenclature.

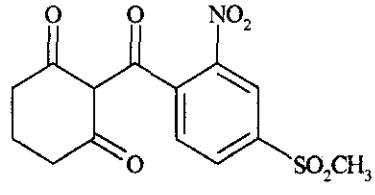
Chemical structure	
Common name	Mesotrione

TABLE A.1. Test Compound Nomenclature.	
Company experimental name	ZA1296
IUPAC name	2-(4-mesyl-2-nitrobenzoyl)cyclohexane-1,3-dione
CAS name	2-[4-(methylsulfonyl)-2-nitrobenzoyl]-1,3-cyclohexanedione
CAS registry number	104206-82-8
End-use product (EP)	4 lb/gal flowable concentrate formulation (Callisto™ Herbicide; EPA Reg. No. 100-1131)

TABLE A.2. Physicochemical Properties of the Technical Grade Test Compound.		
Parameter	Value	Reference
Melting point/range	148.7-152.5 °C	RD Memo, D263245, 2/24/00, H. Podall
pH	3.4 (1% dispersion in water; 25 °C)	RD Memo, D263245, 2/24/00, H. Podall
Density	1.46 g/mL, 20 °C	RD Memo, D263245, 2/24/00, H. Podall
Water solubility	20 ° C 160 ppm, unbuffered water 0.22 g/100 mL, pH 4.8 1.5 g/100mL, pH 6.9 2.2 g/100 mL, pH 9	RD Memo, D263245, 2/24/00, H. Podall
Solvent solubility	20 ° C 0.37 g/100 mL, methanol 1.7 g/100 mL, ethyl acetate 0.27 g/100 mL, toluene 10.4 g/100 mL, acetonitrile <0.03 g/100 mL, heptane 8.1 g/100 mL, acetone	RD Memo, D263245, 2/24/00, H. Podall
Vapor pressure	4.3×10^{-8} torr, 20 °C	RD Memo, D263245, 2/24/00, H. Podall
Dissociation constant, pK_a	3.12, 20 °C	RD Memo, D263245, 2/24/00, H. Podall
Octanol/water partition coefficient, $Log(K_{OW})$	20 ° C log P_{OW} = 0.11 in unbuffered water log P = 0.90 in pH 5 buffer log P <-1 at pH 7 and 9 buffered water	RD Memo, D263245, 2/24/00, H. Podall
UV/visible absorption spectrum	Absorption maximum in methanol at 256 mu, with a molar extinction coefficient of 2.24×10^4 M cm.	RD Memo, D263245, 2/24/00, H. Podall

B. MATERIALS AND METHODS

B.1. Data-Gathering Method

Not applicable to the current submission.

B.2. Enforcement Method

The petitioner has submitted a confirmatory method for determination of residues of mesotrione and its metabolite MNBA in crop samples, LC/MS/MS Method RAM 366/01.

B.2.1. Principle of the Method:

Samples are extracted with acetonitrile/water, and an aliquot of the extract is cleaned up by solid phase extraction (SPE). Residues of mesotrione and MNBA are dissolved in water/methanol and analyzed by HPLC with MS/MS detection.

TABLE B.2.1. Summary Parameters for the Analytical Confirmatory Enforcement Method Used for the Quantitation of Mesotrione Residues in Crop Samples.	
Method ID	RAM 366/01
Analytes	Mesotrione and MNBA
Extraction solvent/technique	Crop samples are to be prepared using an approved method of sample preparation (not described in method). Samples are extracted with acetonitrile:water (50:50, v:v) in the presence of sodium chloride. The volume of extraction solution used is dependent on the water content of the sample. The extract is isolated by centrifugation.
Cleanup strategies	An aliquot is diluted with water and formic acid and cleaned up by SPE (*Oasis* HLB cartridge). Residues are eluted with methanol:formic acid (98:2, v:v). The eluate is evaporated to dryness and redissolved in methanol:water (1:9, v:v) for analysis.
Instrument/Detector	HPLC using a PLRP or PolymerX RP-1 column and a gradient mobile phase of acetonitrile and 0.2% formic acid in water, with triple quadrupole MS/MS detection in the negative ion mode. Deprotonated molecular ions for each analyte (mesotrione and MNBA) are selected and further fragmented. The most abundant daughter ions (291 m/z for mesotrione and 142 m/z for MNBA) are monitored for quantitative analysis.
Standardization method	External standardization using a single standard injected before the samples and reinjected after a maximum of 4 samples. The average peak area for the bracketing standards is used to calculate residues in the sample. It is recommended in the method that matrix-containing standards be used if matrix effects are found to interfere with analysis.
Stability of std solutions	Standards are prepared in methanol and are to be stored at ≤7 °C. The petitioner stated that standards have been shown to be stable in methanol for 22 months, but recommended that fresh standards be prepared every 4 months.
Retention times	Mesotrione: ~3.2 min MNBA: ~1.8 min (based on example chromatograms)

C. RESULTS AND DISCUSSION

C.1. Data-Gathering Method

Not applicable to the current submission.

C.2. Enforcement Method

The petitioner has submitted method validation data for a confirmatory enforcement method. Samples of corn whole plant and corn seed were fortified with mesotrione and MNBA at 0.01, 0.1, and 1.0 ppm, and samples of corn whole plant were additionally fortified at 10 ppm. The results of the method validation study are presented in Table C.2.1.

TABLE C.2.1. Recovery Results from Method Validation of Corn Matrices using the Confirmatory Enforcement Analytical Method.[1]

Matrix	Analyte	Spiking Level (ppm)	Recoveries Obtained	Mean Recovery ± SD [CV]
Corn, whole plant	Mesotrione	0.01	88, 94, 95, 99,100	98 ± 7.2 [7.4]
		0.1	95, 100, 100, 101, 102	
		1.0	82, 85, 89, 97, 102	
		10	99, 104, 104, 108, 109	
	MNBA	0.01	73, 74, 80, 80, 84	87 ± 8.5 [9.7]
		0.1	86, 90, 90, 91, 96	
		1.0	75, 77, 86, 92, 93	
		10	91, 94, 98, 99, 99	
Corn, seed (kernel)	Mesotrione	0.01	71, 82, 85, 94, 96	89 ± 8.4 [9.5]
		0.1	76, 83, 84, 89, 90	
		1.0	93, 93, 93, 96, 103	
	MNBA	0.01	70, 76, 77, 83, 98	90 ± 9.3 [10.4]
		0.1	88, 92, 93, 98, 99	
		1.0	90, 93, 93, 99, 99	

[1] Calibration standards were prepared in the crop matrix, and fortification standard solutions were prepared in methanol.

The petitioner presented data demonstrating that the initial extracts of corn whole plant and kernel were stable for 29-40 days at <7 °C and that the final extracts (in 90:10 water:methanol) were stable at the same temperatures for 6-12 days. In addition, the final extracts were shown to be stable when stored for 4 days at <7 °C and 4 days at ambient temperatures.

TABLE C.2.2. Characteristics for the Enforcement Analytical Method Used for the Quantitation of Mesotrione Residues in Crop Samples.

Analytes	Mesotrione and MNBA
Equipment ID	Agilent 1100 series HPLC; PLRP or PolymerX RP-1 column; API 3000 MS/MS detector
LOQ	0.01 for each analyte
LOD	0.002 ppm for mesotrione and 0.005 ppm for MNBA
Accuracy/Precision	Percent recoveries and coefficients of variance (CVs) indicate acceptable accuracy/precision at the LOQ, 10x LOQ, and 100x LOQ for mesotrione and MNBA from corn whole plant and kernel, and at 1000xLOQ for mesotrione and MNBA from corn whole plant. Recovery ranges (and CVs) from these matrices were 71-109% (9.5%) for mesotrione and 70-99% (10%) for MNBA. See Table C.2.1 above.
Reliability of the Method/ [ILV]	An independent laboratory method validation [ILV] of method RAM 366/01 was conducted to verify the reliability of the method for the determination of residues of mesotrione and MNBA using samples of corn grain, stover, and silage. The values obtained are indicative that method RAM 366/01 is reliable; see Section C.3.
Linearity	The method/detector response was reported to be linear within the range of 0.0008-0.12 µg mL^{-1} (matrix standards) and 0.0001-0.1 µg mL^{-1} (solvent standards); correlation coefficients, r^2 = 0.9977-0.9999.

TABLE C.2.2. Characteristics for the Enforcement Analytical Method Used for the Quantitation of Mesotrione Residues in Crop Samples.	
Specificity	Residues of mesotrione and MNBA in the control samples were <30% LOQ. Peaks were well defined and symmetrical.

C.3. Independent Laboratory Validation

An independent laboratory validation (ILV) of Method RAM 366/01 was conducted by Enviro-Test Laboratories (Edmonton, AB) using samples of field corn grain, stover, and silage (MRID 45651802). Samples of untreated corn grain, stover, and silage, provided to the ILV laboratory by Syngenta, were fortified with mesotrione and MNBA at 0.01 ppm (LOQ), 0.02 ppm, and 0.10 ppm. Fortified and unfortified (control) samples were analyzed using LC/MS/MS Method RAM 366/01 as described in Table B.2.1.

The first ILV trial was successful for corn grain, but low recoveries of both analytes were observed in corn stover and silage in the first trial. After discussion with Syngenta, the laboratory subjected the corn stover and silage extracts to a second SPE cleanup, which did not improve the recoveries. The cleaned up extracts were then diluted (1:10) and reinjected; adequate recoveries were obtained from the diluted extracts (Trial 2). The recoveries of mesotrione and MNBA are reported in Table C.3.1. Residues of mesotrione and MNBA were each below the LOQ (<0.01 ppm) in/on two samples each of unfortified corn grain, stover, and silage. Calibration curves were generated using standards in solvent and crop matrix, which demonstrated good linearity.

The laboratory reported that a set of 14 samples could be extracted in 5-7 hours; LC/MS/MS analysis required approximately 3 hours. The laboratory recommended that extracts be diluted to a final volume of 5-10 mL and recommended a smaller injection volume to reduce matrix effects. The laboratory noted two critical steps: removal of water during SPE cleanup and complete dissolution of extracts in methanol after SPE cleanup.

TABLE C.3.1. Recovery Results Obtained by an Independent Laboratory Validation of the Confirmatory Enforcement Method for the Determination of Residues of Mesotrione and MNBA in Crop Samples.

Matrix	Analyte	Spiking Level (ppm)	Recoveries Obtained	Mean Recovery ± SD [CV]
Corn grain	Mesotrione	0.01	82, 98, 99, 100, 106	94 ± 11 [12]
		0.02	72, 108	
		0.10	86, 87, 88, 91, 108	
	MNBA	0.01	72, 73, 74, 74, 77	73 ± 2.1 [2.9]
		0.02	73, 73	
		0.10	70, 71, 73, 73, 76	

TABLE C.3.1. Recovery Results Obtained by an Independent Laboratory Validation of the Confirmatory Enforcement Method for the Determination of Residues of Mesotrione and MNBA in Crop Samples.

Matrix	Analyte	Spiking Level (ppm)	Recoveries Obtained	Mean Recovery ± SD [CV]
Corn stover	Mesotrione	0.01	76, 86, 92, 109, 111	89 ± 14 [15] [1]
		0.02	81, 105	
		0.10	70, 77, 80, 84, 98	
	MNBA	0.01	72, 81, 88, 92, 105	85 ± 10 [12] [1]
		0.02	93, 97	
		0.10	73, 79, 79, 81, 84	
Corn silage	Mesotrione	0.01	81, 91, 95, 101, 107	94 ± 8.6 [9.1]
		0.02	97, 103	
		0.10	83, 84, 85, 96, 101	
	MNBA	0.01	70, 82, 82, 84, 93	79 ± 6.1 [7.7]
		0.02	78, 83	
		0.10	74, 74, 76, 76, 77	

[1] Calculated by the study reviewer; the values reported in the submission were incorrect.

D. CONCLUSION

The submitted LC/MS/MS method is adequate for use as a confirmatory method for the existing enforcement method for plant commodities. Adequate method validation and independent laboratory validation data were submitted for corn matrices. The method will be forwarded to FDA for inclusion in PAM Volume II as a confirmatory method. Validation by EPA/ACB is not required for this method.

E. REFERENCES

DP Barcodes: D245477 and D260267
Subject: PP#: 8F04954. Mesotrione in/on Field Corn. Evaluation of Residue Data and Analytical Methods. PC Code: 122990.
From: S. Levy
To: J. Stone/J. Tompkins
Date: 6/6/01
MRIDs: 44505118, 44505212-23, 44537109-12, 44901719, and 44942401-03

F. DOCUMENT TRACKING

RDI:
Petition Number(s): PP#2F06443
DP Barcode(s): D283827
PC Code: 122990

Template Version September 2003

Mesotrione/122990/Syngenta Crop Protection
DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3
Crop Field Trial - Sweet Corn

Primary Evaluator	Dynamac Corp	Date: 1/12/05
Approved by	William Cutchin, Chemist TRB/RD	

This DER was originally prepared under contract by Dynamac Corporation. The DER has been reviewed by TRB and revised to reflect current OPP policies.

STUDY REPORT:

45651801 Cobin, J. (2002) Mesotrione - Magnitude of the Residues in or on Sweet Corn: Laboratory Study Number: 487-01. Unpublished study prepared by Syngenta Crop Protection, Inc. 251 p.

EXECUTIVE SUMMARY:

Syngenta Crop Protection, Inc. has submitted crop field trial data for sweet corn. A total of twelve sweet corn field trials were conducted during the 2001 growing season in Regions I (NY and PA), II (NC), III (FL), V (IL, MI, MN, OH, and WI), X (CA), XI (ID), and XII (WA). The number and locations of field trials are in accordance with OPPTS Guideline 860.1500 for sweet corn.

A single preemergence application of the 4 lb/gal suspension concentrate formulation (equivalent to a flowable concentrate; FlC) was made at ~0.270 lb ai/A to the soil surface preplant (on the day of planting) or within 2 days after planting of sweet corn, followed, 43-116 days later, by a single postemergence foliar application of the 4 lb/gal FlC formulation at ~0.16 lb ai/A. Total application rates (pre- and postemergence) ranged 0.428-0.451 lb ai/A. Sweet corn forage (with ears) was harvested 14 days PHI, forage (without ears) and ears were harvested 26-31 days PHI, and stover was harvested 52-71 days PHI. Additional samples of forage (with and without ears) and ears were collected from two field trials to evaluate residue decline. The petitioner stated (in the summary of the field trial procedures) that adjuvants [SOC or COC at 1% (v:v) and AMS or UAN at 2.5% (v:v)] were added to the spray mixtures for postemergence applications. However, no information pertaining to spray adjuvants was included in the field trial summaries (field trial raw data).

Samples of sweet corn commodities were analyzed for residues of mesotrione and its metabolite MNBA using Method TMR0882B, the current enforcement method for field corn commodities which is an HPLC method with fluorescence detection. The validated limit of quantitation (LOQ) was 0.01 ppm for each metabolite in sweet corn forage, ears, and stover. This method is adequate for data collection based on acceptable concurrent method recovery data.

The maximum storage intervals of sweet corn samples from harvest to analysis were 220 days (7.2 months) for forage, 209 days (6.9 months) for ears, and 169 days (5.6 months) for stover.

Adequate storage stability data were previously submitted (PP#8F04954; DP Barcodes D245477 and D260267, 6/6/01, S. Levy) which demonstrated that residues of mesotrione and MNBA are stable under frozen storage conditions for up to 42 months in/on field corn forage, grain, and stover. These data will support the storage conditions and intervals of samples from the submitted sweet corn field trials.

Residues of mesotrione were <0.01-0.879 ppm in/on sweet corn forage (with ears) harvested 14 days PHI, <0.01-0.396 ppm in/on sweet corn forage (without ears) harvested 26-31 days PHI, <0.01 ppm in/on sweet corn ears harvested 26-31 days PHI, and <0.01-1.204 ppm in/on sweet corn stover harvested 52-71 days PHI. Residues of MNBA were <0.01-0.061 ppm in/on sweet corn forage (with ears), <0.01-0.037 ppm in/on sweet corn forage (without ears), <0.01 ppm in/on sweet corn ears, and <0.01-0.075 ppm in/on sweet corn stover.

We note that residues of mesotrione were at or below the LOQ (≤0.01 ppm) in/on samples of forage (without ears) collected 26-31 days PHI from all field trials except two (CA and ID), in/on ear samples from all field trials, and in/on stover samples from all field trials except one (CA). In the ID field trial, residues of mesotrione were <0.01 and 0.052 ppm in/on forage (without ears) harvested 30 days PHI. In the CA field trial, residues of mesotrione were much higher than in the other trials; residues were 0.381 and 0.396 ppm in/on forage (without ears) harvested 30 days PHI, and residues were 0.949 and 1.204 ppm in/on stover harvested 58 days PHI. Residues of mesotrione were quantifiable, ranging 0.017-0.879 ppm, in/on forage (with ears) samples harvested 14 days PHI in four of the field trials, and nonquantifiable (<0.01 ppm) in/on all forage (with ears) samples from the other eight field trials.

Data from the residue decline studies demonstrate that residues of mesotrione and MNBA decline in forage (with and without ears) with increasing harvest intervals. In one residue decline trial (CA), average residues of mesotrione and MNBA in/on forage (with ears) declined from 4.63 and 0.093 ppm, respectively, at the 0-day sampling interval, to 0.807 and 0.054 ppm, respectively, at the 14-day sampling interval. Average residues of mesotrione and MNBA in/on forage (without ears) declined from 0.398 and 0.039 ppm, respectively, at the 23-day sampling interval, to 0.281 and 0.030 ppm, respectively, at the 37-day sampling interval. In the second residue decline trial (MI), average residues of mesotrione and MNBA in/on forage (with ears) declined from 3.53 and 0.048 ppm, respectively, at the 0-day sampling interval, to <0.01 ppm (both analytes) at the 14-day sampling interval; residues in/on forage (without ears) were below the LOQ in/on all samples from this decline study. Residues in sweet corn ears were below the LOQ in all samples from both decline studies.

STUDY/WAIVER ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS:

Under the conditions and parameters used in the study, the sweet corn field trial residue data are classified as scientifically.

The acceptability of this study for regulatory purposes is addressed in the forthcoming U.S. EPA Residue Chemistry Summary Document, DP Barcode D283827.

COMPLIANCE:

Signed and dated GLP, Quality Assurance and Data Confidentiality statements were provided. No deviations from regulatory requirements were reported which would impact the validity of the study.

A. BACKGROUND INFORMATION

Mesotrione is a triketone herbicide which inhibits the enzyme p-hydroxyphenylpyruvate dioxygenase (HPPD), disrupting carotenoid biosynthesis. This process leads to the destruction of chlorophyll, resulting in a bleaching effect in susceptible plants. Mesotrione is intended for preemergence and postemergence use for selective control of annual broadleaf weeds.

Mesotrione is currently registered for use on field and pop corn. The petitioner is now proposing uses on sweet corn.

TABLE A.1. Test Compound Nomenclature.

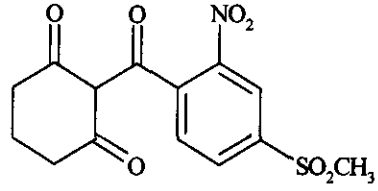
Chemical structure	
Common name	Mesotrione
Company experimental name	ZA1296
IUPAC name	2-(4-mesyl-2-nitrobenzoyl)cyclohexane-1,3-dione
CAS name	2-[4-(methylsulfonyl)-2-nitrobenzoyl]-1,3-cyclohexanedione
CAS registry number	104206-82-8
End-use product (EP)	4 lb/gal flowable concentrate formulation (Callisto™ Herbicide; EPA Reg. No. 100-1131)

TABLE A.2. Physicochemical Properties of the Technical Grade Test Compound.

Parameter	Value	Reference
Melting point/range	148.7-152.5 °C	RD Memo, D263245, 2/24/00, H. Podall
pH	3.4 (1% dispersion in water; 25 °C)	RD Memo, D263245, 2/24/00, H. Podall
Density	1.46 g/mL, 20 °C	RD Memo, D263245, 2/24/00, H. Podall
Water solubility	20 ° C 160 ppm, unbuffered water 0.22 g/100 mL, pH 4.8 1.5 g/100mL, pH 6.9 2.2 g/100 mL, pH 9	RD Memo, D263245, 2/24/00, H. Podall

TABLE A.2. Physicochemical Properties of the Technical Grade Test Compound.		
Parameter	Value	Reference
Solvent solubility	20 ° C 0.37 g/100 mL, methanol 1.7 g/100 mL, ethyl acetate 0.27 g/100 mL, toluene 10.4 g/100 mL, acetonitrile <0.03 g/100 mL, heptane 8.1 g/100 mL, acetone	RD Memo, D263245, 2/24/00, H. Podall
Vapor pressure	4.3 x 10^{-8} torr, 20 °C	RD Memo, D263245, 2/24/00, H. Podall
Dissociation constant, pK_a	3.12, 20 °C	RD Memo, D263245, 2/24/00, H. Podall
Octanol/water partition coefficient, Log(K_{ow})	20 ° C log P_{ow} = 0.11 in unbuffered water log P = 0.90 in pH 5 buffer log P <-1 at pH 7 and 9 buffered water	RD Memo, D263245, 2/24/00, H. Podall
UV/visible absorption spectrum	Absorption maximum in methanol at 256 mu, with a molar extinction coefficient of 2.24 x 10^4 M cm.	RD Memo, D263245, 2/24/00, H. Podall

B. EXPERIMENTAL DESIGN

B.1. Study Site Information

TABLE B.1.1. Trial Site Conditions.						
Trial Identification (City, State; Year)	Soil characteristics				Meteorological data	
	Type	%OM	pH	CEC (meq/g)	Range of monthly total rainfall (inches)	Range of monthly average temperatures (°F)
Delavan, WI; 2001	silt loam	Not provided			2.23-7.21 (also received overhead irrigation)	52.1-75.6
Mt. Vernon, WA; 2001	silt loam	Not provided			0.82-6.00 (information not available for all months)	49.8-63.3 (information not available for all months)
Visalia, CA; 2001	sandy loam	Not provided			0.0-0.48 (information not available for all months; also received furrow irrigation)	67.9-80.3 (information not available for all months)
Rose Hill, NC; 2001	loamy sand	Not provided			0.60-6.34 (also received overhead irrigation)	61.5-78.4
New Holland, OH; 2001	silt loam	Not provided			1.53-4.26	64.3-73.9 (information not available for all days)
North Rose, NY; 2001	sand	Not provided			1.49-4.32	48.3-73.5
Greenleaf, ID; 2001	sandy loam	Not provided			0.0-0.47 (also received furrow irrigation)	61.9-75.0
Champaign, IL; 2001	silt loam	Not provided			2.83-4.79 (also received overhead irrigation)	64.2-75.5
Germansville, PA; 2001	loam	Not provided			1.59-5.55 (also received drip irrigation)	36.5-73.7

TABLE B.1.1. Trial Site Conditions.

Trial Identification (City, State; Year)	Soil characteristics				Meteorological data	
	Type	%OM	pH	CEC (meq/g)	Range of monthly total rainfall (inches)	Range of monthly average temperatures (°F)
Vero Beach, FL; 2001	sand	Not provided			6.52-11.33 (information not available for all months; also received overhead irrigation)	71.1-77.5 (information not available for all months)
Conklin, MI; 2001	loam	Not provided			1.66-6.09 (also received overhead irrigation)	49.5-70.3
Geneva, MN; 2001	loam	Not provided			1.41-7.82	47.1-73.7

Information concerning maintenance chemicals and weather conditions (total monthly rainfall and average monthly temperature, as well as comparison of the conditions to historical averages) were provided for each field trial. No unusual weather conditions were noted which may have affected the study results.

TABLE B.1.2. Study Use Pattern.

Location (City, State; Year)	EP[1]	Application					Tank Mix Adjuvants
		Method; Timing	Vol. (GPA[2])	Rate (lb ai/A)	RTI[3] (days)	Total Rate (lb ai/A)	
Delavan, WI; 2001	4 lb/gal FlC	1: Broadcast; Preemergence one day after planting	10.23	0.270	47	0.428	None specified
		2: Foliar broadcast; VT crop growth stage (tasseling)	17.9	0.158			None specified [4]
Mt. Vernon, WA; 2001	4 lb/gal FlC	1: Broadcast; Post plant, on the day of planting	16.49	0.270	116	0.430	None specified
		2: Foliar broadcast; Pollen shed crop growth stage	19.91	0.159			None specified [4]
Visalia, CA; 2001	4 lb/gal FlC	1: Broadcast; Crop growth stage 01, one day after planting	19.53	0.268	69	0.431	None specified
		2: Foliar broadcast; Crop growth stage 59	18.87	0.162			None specified [4]
Rose Hill, NC; 2001	4 lb/gal FlC	1: Broadcast; The day of planting	15.16	0.275	57	0.438	None specified
		2: Foliar broadcast; Tassle crop growth stage (55)	19.05	0.163			None specified [4]
New Holland, OH; 2001	4 lb/gal FlC	1: Broadcast; Two days after planting	15.75	0.280	48	0.450	None specified
		2: Foliar broadcast; Crop growth stage not reported	16.53	0.170			None specified [4]
North Rose, NY; 2001	4 lb/gal FlC	1: Broadcast; At planting	17.87	0.272	61	0.434	None specified
		2: Foliar broadcast; 8-9 leaf crop growth stage	18.43	0.162			None specified [4]

TABLE B.1.2. Study Use Pattern.

Location (City, State; Year)	EP[1]	Application					Tank Mix Adjuvants
		Method; Timing	Vol. (GPA[2])	Rate (lb ai/A)	RTI[3] (days)	Total Rate (lb ai/A)	
Greenleaf, ID; 2001	4 lb/gal FlC	1: Broadcast; Pre-plant, on the day of planting	19.97	0.270	43	0.430	None specified
		2: Foliar broadcast; 30 days prior to harvest	19.81	0.160			None specified [4]
Champaign, IL; 2001	4 lb/gal FlC	1: Broadcast; on the day of planting	14.49	0.268	43	0.434	None specified
		2: Foliar broadcast; Crop growth stage 34	15.97	0.166			None specified [4]
Germansville, PA; 2001	4 lb/gal FlC	1: Broadcast; Two days after planting	19.27	0.285	63	0.451	None specified
		2: Foliar broadcast; Early tassel/ear formation	18.85	0.166			None specified [4]
Vero Beach, FL; 2001	4 lb/gal FlC	1: Broadcast; Just planted	16.39	0.279	50	0.437	None specified
		2: Foliar broadcast; Tassel visible	17.2	0.158			None specified [4]
Conklin, MI; 2001	4 lb/gal FlC	1: Broadcast; Preemergence, on the day of planting	17.17	0.273	56	0.434	None specified
		2: Foliar broadcast; VT crop growth stage	14.86	0.161			None specified [4]
Geneva, MN; 2001	4 lb/gal FlC	1: Broadcast; On the day of planting	10.57	0.271	50	0.439	None specified
		2: Foliar broadcast; Tassel initiation	16.83	0.168			None specified [4]

[1] EP = End-use Product
[2] GPA = Gallons per acre
[3] RTI = Retreatment Interval
[4] The individual field trial summaries did not provide any indication that adjuvants were used; however, the summary of the field procedures stated that postemergence foliar applications were made with SOC or COC, at 1% v:v, and AMS or UAN, at 2.5% v:v, added to the spray solution.

TABLE B.1.3. Trial Numbers and Geographical Locations.

NAFTA Growing Region	Sweet corn	
	Submitted	Requested [1] US
1	2	2
1A		
2	1	1
3	1	1
4		
5	5	5
5A		

TABLE B.1.3. Trial Numbers and Geographical Locations.		
NAFTA Growing Region	Sweet corn	
	Submitted	Requested [1] US
5B		
6		
7		
7A		
8		
9		
10	1	1
11	1	1
12	1	1
13		
14		
15		
16		
17		
18		
19		
20		
21		
Total	12	12

[1] OPPTS 860.1500, Table 5.

B.2. Sample Handling and Preparation

A single untreated and duplicate treated samples of forage (with ears), forage (without ears), ears, and stover were collected at each trial site. Stover samples were collected from stalks which had been dried down to approximately 80-85% dry matter. The length of time between sample collection and transfer to frozen storage was not specified. Samples were shipped frozen (method of shipment not specified) within 84 days of collection to Syngenta for residue analysis. Samples were stored frozen (temperature unspecified) at Syngenta prior to residue analysis. The method of preparation of samples prior to analysis (homogenization, etc.) was not specified; the petitioner instead referenced Syngenta SOPs for sample preparation which were not available to the study reviewer.

We note that the petitioner has referred to sweet corn ears throughout their submission; we assume that "ears" are representative of kernels plus cobs with husks removed (K+CWHR), the regulated RAC of sweet corn.

B.3. Analytical Methodology

Samples of sweet corn commodities were analyzed for residues of mesotrione and its metabolite MNBA using Method TMR0882B, an HPLC method with fluorescence detection (FLD) which is the current enforcement method for field corn commodities. A method description was not included for the sweet corn field trials; however, the method has been reviewed by HED (PP#8F04954, DP Barcodes D245477 and D260267, 6/6/01, S. Levy) and has undergone validation by ACB. The validated LOQ was 0.01 ppm each for residues of mesotrione and MNBA in/on sweet corn ears, forage (with and without ears), and stover. No limit of detection (LOD) was reported.

C. RESULTS AND DISCUSSION

Sample storage conditions and intervals are summarized in Table C.2. The maximum storage intervals of sweet corn samples from harvest to analysis were 220 days (7.2 months) for forage, 209 days (6.9 months) for ears, and 169 days (5.6 months) for stover. Adequate storage stability data were previously submitted (PP#8F04954, DP Barcodes D245477 and D260267, 6/6/01, S. Levy) which demonstrated that residues of mesotrione and MNBA are stable under frozen storage conditions for up to 42 months in/on field corn forage, grain, and stover. These data support the storage conditions and intervals of samples from the submitted sweet corn field trials.

Concurrent method recovery data are presented in Table C.1. Samples of sweet corn commodities were analyzed for residues of mesotrione and its metabolite MNBA using HPLC/FLD Method TMR0882B, the current enforcement method for field corn commodities. The validated LOQ was 0.01 ppm each for residues of mesotrione and MNBA in/on sweet corn ears, forage (with and without ears), and stover. This method is adequate for data collection based on acceptable concurrent method recovery data. Apparent residues of mesotrione and MNBA were each below the LOQ in/on 16 samples each of untreated sweet corn forage with ears, forage without ears, and ears, and in/on 12 samples of sweet corn stover.

Residue data from the sweet corn field trials are reported in Table C.3. A summary of residue data in sweet corn following treatment with the 4 lb/gal FlC formulation is presented in Table C.4. Following pre- and postemergence applications of the 4 lb/gal FlC formulation at total rates of 0.428-0.451 lb ai/A, residues of mesotrione were <0.01-0.879 ppm in/on sweet corn forage (with ears) harvested 14 days PHI, <0.01-0.396 ppm in/on sweet corn forage (without ears) harvested 26-31 days PHI, <0.01 ppm in/on sweet corn ears harvested 26-31 days PHI, and <0.01-1.204 ppm in/on sweet corn stover harvested 52-71 days PHI. Residues of MNBA were <0.01-0.061 ppm in/on sweet corn forage (with ears), <0.01-0.037 ppm in/on sweet corn forage (without ears), <0.01 ppm in/on sweet corn ears, and <0.01-0.075 ppm in/on sweet corn stover.

We note that residues of mesotrione were at or below the LOQ (≤0.01 ppm) in/on samples of forage (without ears) collected 26-31 days PHI from all field trials except two (CA and ID), in/on ear samples from all field trials, and in/on stover samples from all field trials except one (CA). In the ID field trial, residues of mesotrione were <0.01 and 0.052 ppm in/on forage (without ears)

harvested 30 days PHI. In the CA field trial, residues of mesotrione were much higher than in the other trials; residues were 0.381 and 0.396 ppm in/on forage (without ears) harvested 30 days PHI, and residues were 0.949 and 1.204 ppm in/on stover harvested 58 days PHI. Residues of mesotrione were quantifiable, ranging 0.017-0.879 ppm, in/on forage (with ears) samples harvested 14 days PHI in four of the field trials, and nonquantifiable (<0.01 ppm) in/on all forage (with ears) samples from the other eight field trials.

Data from the residue decline studies demonstrate that residues of mesotrione and MNBA decline in forage (with and without ears) with increasing harvest intervals. In one residue decline trial (CA), average residues of mesotrione and MNBA in/on forage (with ears) declined from 4.63 and 0.093 ppm, respectively, at the 0-day sampling interval, to 0.807 and 0.054 ppm, respectively, at the 14-day sampling interval. Average residues of mesotrione and MNBA in/on forage (without ears) declined from 0.398 and 0.039 ppm, respectively, at the 23-day sampling interval, to 0.281 and 0.030 ppm, respectively, at the 37-day sampling interval. In the second residue decline trial (MI), average residues of mesotrione and MNBA in/on forage (with ears) declined from 3.53 and 0.048 ppm, respectively, at the 0-day sampling interval, to <0.01 ppm (both analytes) at the 14-day sampling interval; residues in/on forage (without ears) were below the LOQ in/on all samples from this decline study. Residues in sweet corn ears were below the LOQ in all samples from both decline studies.

A total of twelve sweet corn field trials were conducted during the 2001 growing season in Regions I (NY and PA), II (NC), III (FL), V (IL, MI, MN, OH, and WI), X (CA), XI (ID), and XII (WA). The number and locations of field trials are in accordance with OPPTS Guideline 860.1500 for sweet corn.

TABLE C.1. Summary of Concurrent Recoveries of Mesotrione from Sweet Corn Matrices.

Matrix	Analyte	Spike level (ppm)	Sample size (n)	Recoveries (%)	Mean ± std dev
Sweet corn forage, with ears	Mesotrione	0.01	6	54 [1], 68, 72, 84, 85, 94	78 ± 11
		0.10	6	70, 73, 75, 77, 79, 84 [2]	
		5.00	1	93	
	MNBA	0.01	6	70, 73, 73, 78, 79, 96	79 ± 9.2
		0.10	6	65, 71, 75, 80, 84, 90	
		5.00	1	91	
Sweet corn forage, without ears	Mesotrione	0.01	6	68, 69, 82, 95, 104, 107	86 ± 13
		0.10	6	72, 75 [2], 79, 91, 92, 94	
		1.00	3	76, 91, 102	
	MNBA	0.01	8	64, 66, 81, 86, 90, 90, 90, 98	87 ± 13
		0.10	6	68, 78, 89, 93, 93, 119	
		1.00	3	83, 89, 98	

TABLE C.1. Summary of Concurrent Recoveries of Mesotrione from Sweet Corn Matrices.

Matrix	Analyte	Spike level (ppm)	Sample size (n)	Recoveries (%)	Mean ± std dev
Sweet corn ears	Mesotrione	0.01	5	59 [1], 64, 65, 69, 77	71 ± 6.8
		0.10	5	66, 71, 75, 78, 81	
		1.00	1	74	
	MNBA	0.01	4	78, 88, 93, 94	84 ± 12
		0.10	6	63, 65, 87, 90, 93, 97	
		1.00	1	73	
Sweet corn stover	Mesotrione	0.01	4	66, 67, 69, 89 [2]	76 ± 7.9
		0.10	5	65, 76, 77, 78, 78	
		1.00	1	83	
		2.00	1	83	
	MNBA	0.01	5	65, 67, 67, 80, 82	79 ± 11
		0.10	7	68, 73, 75, 76, 81, 90, 94	
		1.00	1	93	
		2.00	1	98	

[1] Corrected for residues in control, which were attributed to possible carryover from the standard injected just prior to the fortified recovery sample.
[2] Average of replicate injections.

TABLE C.2. Summary of Storage Conditions

Matrix	Storage Temp. (°C)	Actual Storage Duration [1]	Limit of Demonstrated Storage Stability
Sweet corn forage, with ears	Frozen; temperature unspecified	110-220 days (3.6-7.2 months)	42 months, for residues of mesotrione and MNBA in/on field corn forage, stover, and grain (PP#8F04954; DP Barcodes D245477 and D260267, 6/6/01, S. Levy)
Sweet corn forage, without ears		108-207 days (3.6-6.8 months)	
Sweet corn ears		94-196 days (3.1-6.4 months)	
Sweet corn stover		49-152 days (1.6-5.0 months)	

[1] Samples were analyzed within 23 days of extraction.

TABLE C.3. Residue Data from Crop Field Trials with Mesotrione.

Trial ID (City, State; Year)	Region	Crop Variety	Total Rate (lb ai/A)	Sweet Corn Commodity	PHI (days)	Residues (ppm)	
						Mesotrione	MNBA
Delavan, WI; 2001	V	NK199	0.428	Forage, with ears	14	<0.01, <0.01	<0.01, <0.01
				Forage, without ears	30	<0.01, 0.01	<0.01, <0.01
				Ears	30	<0.01, <0.01	<0.01, <0.01
				Stover	56	<0.01, <0.01	<0.01, <0.01
Mt. Vernon, WA; 2001	XII	Jubilee	0.430	Forage, with ears	14	0.031 [1], 0.099 [1]	<0.01, <0.01
				Forage, without ears	28	<0.01, <0.01	<0.01, <0.01
				Ears	28	<0.01, <0.01	<0.01, <0.01
				Stover	71	<0.01, <0.01	<0.01, <0.01

TABLE C.3. Residue Data from Crop Field Trials with Mesotrione.

Trial ID (City, State; Year)	Region	Crop Variety	Total Rate (lb ai/A)	Sweet Corn Commodity	PHI (days)	Residues (ppm)	
						Mesotrione	MNBA
Visalia, CA; 2001	X	Silver Queen	0.431	Forage, with ears	0	4.29, 4.96	0.087, 0.098
					7	0.823, 1.28	0.041, 0.065
					14	0.734, 0.879	0.047, 0.061
				Forage, without ears	23	0.348, 0.448	0.035, 0.042
					30	0.381, 0.396	0.033, 0.034
					37	0.233, 0.329	0.018, 0.041
				Ears	23	<0.01, <0.01	<0.01, <0.01
					30	<0.01, <0.01	<0.01, <0.01
					37	<0.01, <0.01	<0.01, <0.01
				Stover	58	0.949 [1], 1.204 [1]	0.075, 0.066
Rose Hill, NC; 2001	II	G90 F1	0.438	Forage, with ears	14	<0.01, 0.019 [1]	<0.01, 0.017
				Forage, without ears	26	<0.01, <0.01	<0.01, <0.01
				Ears	26	<0.01, <0.01	<0.01, <0.01
				Stover	54	<0.01, <0.01	<0.01, <0.01
New Holland, OH; 2001	V	Bodacious	0.450	Forage, with ears	14	<0.01, <0.01	<0.01, <0.01
				Forage, without ears	30	<0.01, <0.01	<0.01, <0.01
				Ears	30	<0.01, <0.01	<0.01, <0.01
				Stover	52	<0.01, <0.01	<0.01, <0.01
North Rose, NY; 2001	I	GH-2783	0.434	Forage, with ears	14	<0.01, <0.01	<0.01, <0.01
				Forage, without ears	30	<0.01, <0.01	<0.01, <0.01
				Ears	30	<0.01, <0.01	<0.01, <0.01
				Stover	67	<0.01, <0.01	<0.01, <0.01
Greenleaf, ID; 2001	XI	Sugar Buns	0.430	Forage, with ears	14	0.017 [1], 0.023 [1]	<0.01, <0.01
				Forage, without ears	30	<0.01, 0.052	<0.01, 0.037
				Ears	30	<0.01, <0.01	<0.01, <0.01
				Stover	61	<0.01, <0.01	<0.01, <0.01
Champaign, IL; 2001	V	Kandy King	0.434	Forage, with ears	14	<0.01, <0.01	<0.01, <0.01
				Forage, without ears	31	<0.01, <0.01	<0.01, <0.01
				Ears	31	<0.01, <0.01	<0.01, <0.01
				Stover	60	<0.01, <0.01	<0.01, <0.01
Germansville, PA; 2001	I	Argent	0.451	Forage, with ears	14	<0.01, <0.01	<0.01, <0.01
				Forage, without ears	30	<0.01, <0.01	<0.01, <0.01
				Ears	30	<0.01, <0.01	<0.01, <0.01
				Stover	70	<0.01, <0.01	<0.01, <0.01

TABLE C.3. Residue Data from Crop Field Trials with Mesotrione.

Trial ID (City, State; Year)	Region	Crop Variety	Total Rate (lb ai/A)	Sweet Corn Commodity	PHI (days)	Residues (ppm)	
						Mesotrione	MNBA
Vero Beach, FL; 2001	III	Silver Queen	0.437	Forage, with ears	14	<0.01, <0.01	<0.01, <0.01
				Forage, without ears	28	<0.01, <0.01	<0.01, <0.01
				Ears	28	<0.01, <0.01	<0.01, <0.01
				Stover	62	<0.01, <0.01	<0.01, <0.01
Conklin, MI; 2001	V	Excellency	0.434	Forage, with ears	0	3.09, 3.96	0.044, 0.052
					7	0.013, 0.015	<0.01, <0.01
					14	<0.01, <0.01	<0.01, <0.01
				Forage, without ears	23	<0.01, <0.01	<0.01, <0.01
					30	<0.01, <0.01	<0.01, <0.01
					36	<0.01, <0.01	<0.01, <0.01
				Ears	23	<0.01, <0.01	<0.01, <0.01
					30	<0.01, <0.01	<0.01, <0.01
					36	<0.01, <0.01	<0.01, <0.01
				Stover	70	<0.01, <0.01	<0.01, <0.01
Geneva, MN; 2001	V	Maple Sweet	0.439	Forage, with ears	14	<0.01, <0.01	<0.01, <0.01
				Forage, without ears	30	<0.01, <0.01	<0.01, <0.01
				Ears	30	<0.01, <0.01	<0.01, <0.01
				Stover	55	<0.01, <0.01	<0.01, <0.01

[1] Highest residue value from duplicate analyses. Analyses were repeated by petitioner to verify residue values; the results of the second analysis were consistent with the initial analysis.

TABLE C.4. Summary of Residue Data from Crop Field Trials with Mesotrione.

Sweet Corn Commodity	Total Applic. Rate (lb ai/A)	PHI (days)	Analyte	Residue Levels (ppm) [1]						
				n	Min.	Max.	HAFT[2]	Median (STMdR[3])	Mean (STMR[4])	Std. Dev.
Forage, with ears	0.428-0.451	14	Mesotrione	24	<0.01	0.879	0.807	0.005	0.079	0.226
			MNBA	24	<0.01	0.061	0.054	0.005	0.010	0.014
Forage, without ears		26-31	Mesotrione	24	<0.01	0.396	0.389	0.005	0.039	0.108
			MNBA	24	<0.01	0.037	0.034	0.005	0.009	0.010
Ears		26-31	Mesotrione	24	<0.01	<0.01	<0.01	0.005	0.005	0.0
			MNBA	24	<0.01	<0.01	<0.01	0.005	0.005	0.0
Stover		52-71	Mesotrione	24	<0.01	1.204	1.077	0.005	0.094	0.305
			MNBA	24	<0.01	0.075	0.071	0.005	0.010	0.019

[1] For the determination of minimum, maximum, and HAFT values, the LOQ (<0.01 ppm) was used for residues reported as <LOQ in Table C.3. For the determination of the median, mean, and standard deviation values, half the LOQ (0.005 ppm) was used for residues reported as <LOQ.
[2] HAFT = Highest Average Field Trial.
[3] STMdR = Supervised Trial Median Residue.
[4] STMR = Supervised Trial Mean Residue.

D. CONCLUSION

The submitted sweet corn field trial data reflect use of mesotrione at a seasonal rate of ~0.43 lb ai/A with a 30-day PHI for forage and ears and a ~60-day PHI for stover. A single preemergence application of the 4 lb/gal FlC formulation was made within 2 days of planting at ~0.27 lb ai/A and a postemergence application was made at ~0.16 lb ai/A approximately 30 days prior to harvest. Acceptable methods were used for quantitation of residues in/on sweet corn commodities. Mesotrione residues on sweet corn treated at a total seasonal rate of 0.428-0.451 lb ai/A did not exceed 0.01 ppm on ears at 26-31 day PHI, 0.81 ppm on forage with ears at 14 day PHI, 0.40 ppm on forage without ears at 26-31 day PHI, and 1.20 ppm on stover at 52-71 day PHI.

E. REFERENCES

DP Barcodes: D245477 and D260267
Subject: PP#: 8F04954. Mesotrione in/on Field Corn. Evaluation of Residue Data and Analytical Methods. PC Code: 122990.
From: S. Levy
To: J. Stone/J. Tompkins
Date: 6/6/01
MRIDs: 44505118, 44505212-44505223, 44537109-44537112, 44901719, and 44942401-44942403

F. DOCUMENT TRACKING

RDI:
Petition Number(s): 2F06443
DP Barcode(s): D283827
PC Code: 122990

Template Version September 2003